EXTERNAL INSULATIONS

Contract NAS9-10583

Final Study Report

September 28, 1971

Colspan, Inc. J. R. Wilson

CR-115203

External Insulation Systems

for

Cryogenic Storage Systems

Contract NAS 9-10583

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September 28, 1971

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ABSTRACT

Success in considering the feasibility of application of multilayer dielectric film reflectors to cryogenic insulation materials technology is outlined. Future requirements for cryogenic thermal protection system performance are considered, and a brief description presented of radiant energy transport processes in such systems as currently understood.

Optical reflector designs which block radiant energy transfer out to 25 μ with high efficiency are discussed. These reflectors are expected to exhibit emissivities substantially lower than those of present materials used for such purposes. A suggested program for further investigation of the properties of these devices is presented.

LIST OF FIGURES

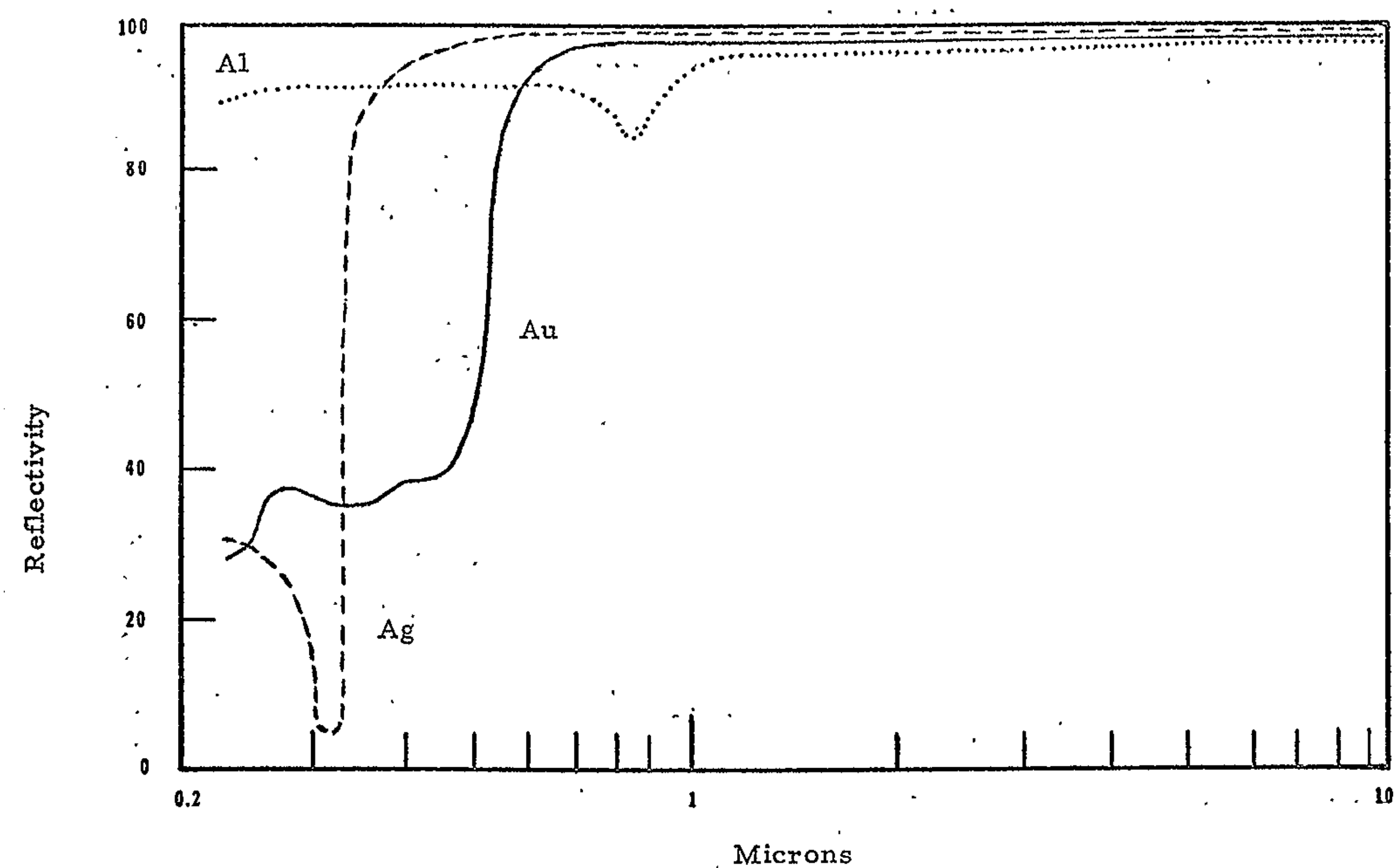


FIGURE 1

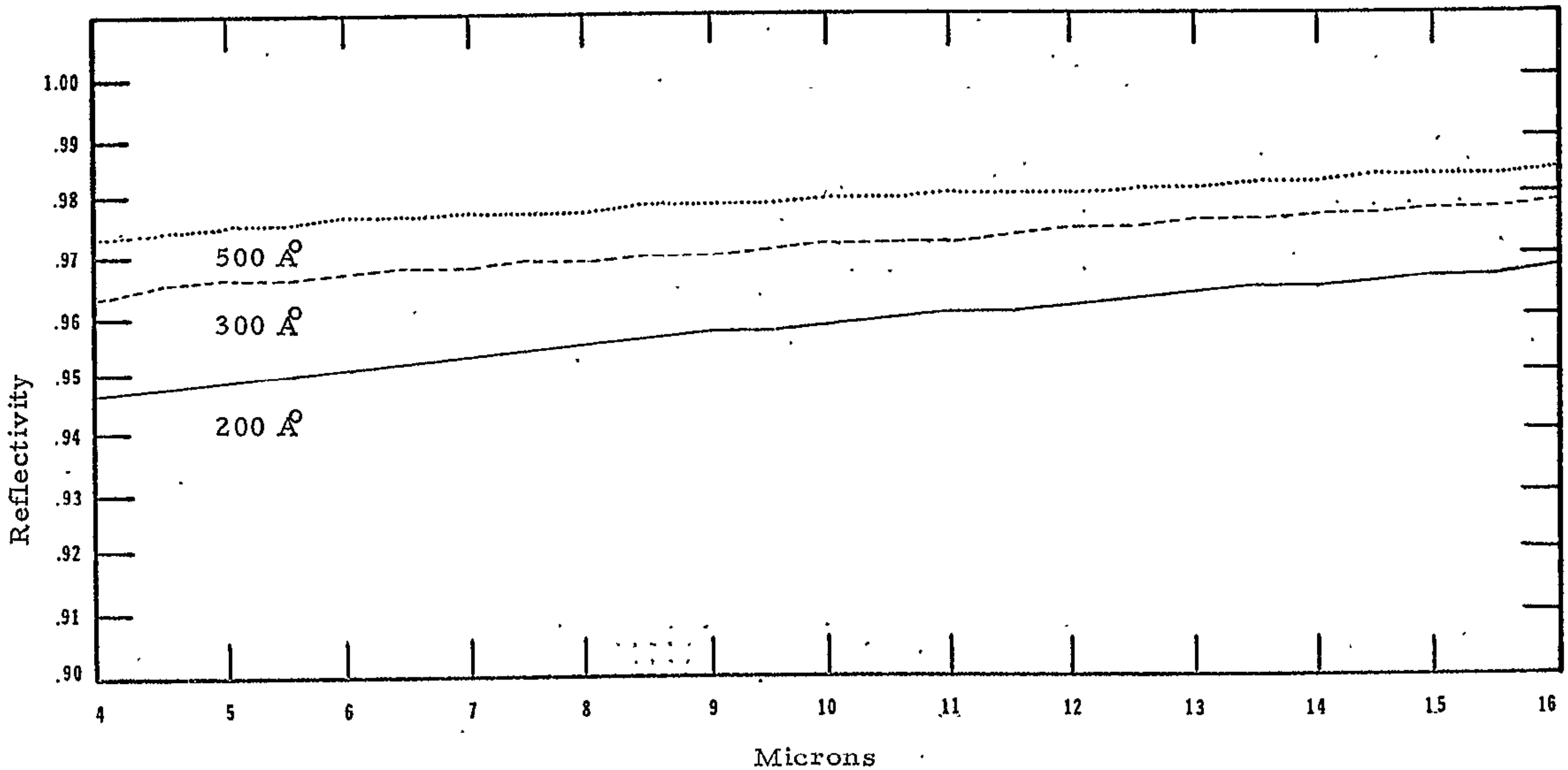
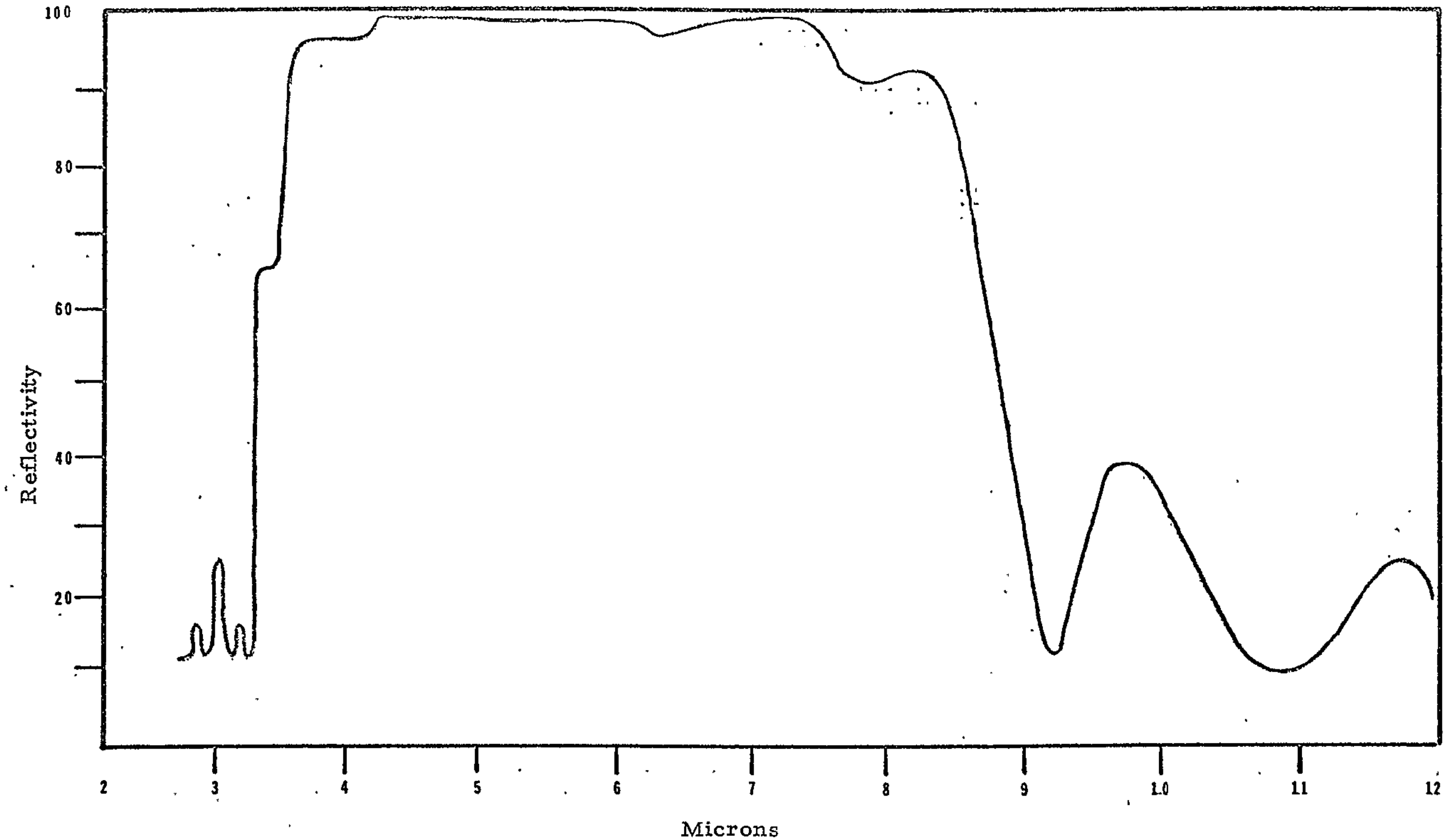

Reflectivity
1.00
.99
.98
.97
.96
.95
.94
.93
.92
.91
.90
500 Å
300 Å
200 Å
4
5
6
7
8
9
10
11
12
13
14
15
16
Microns

FIGURE 2



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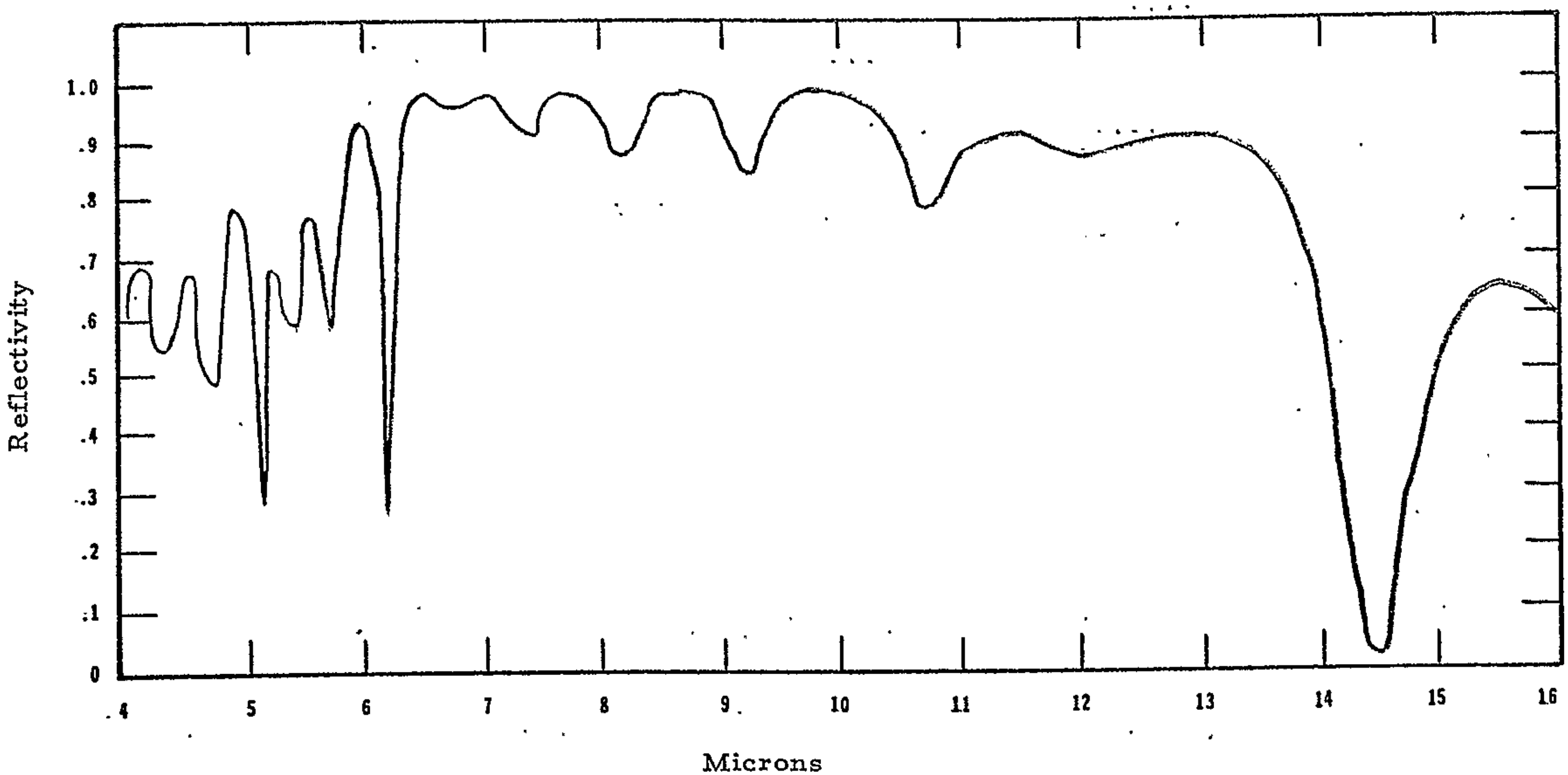
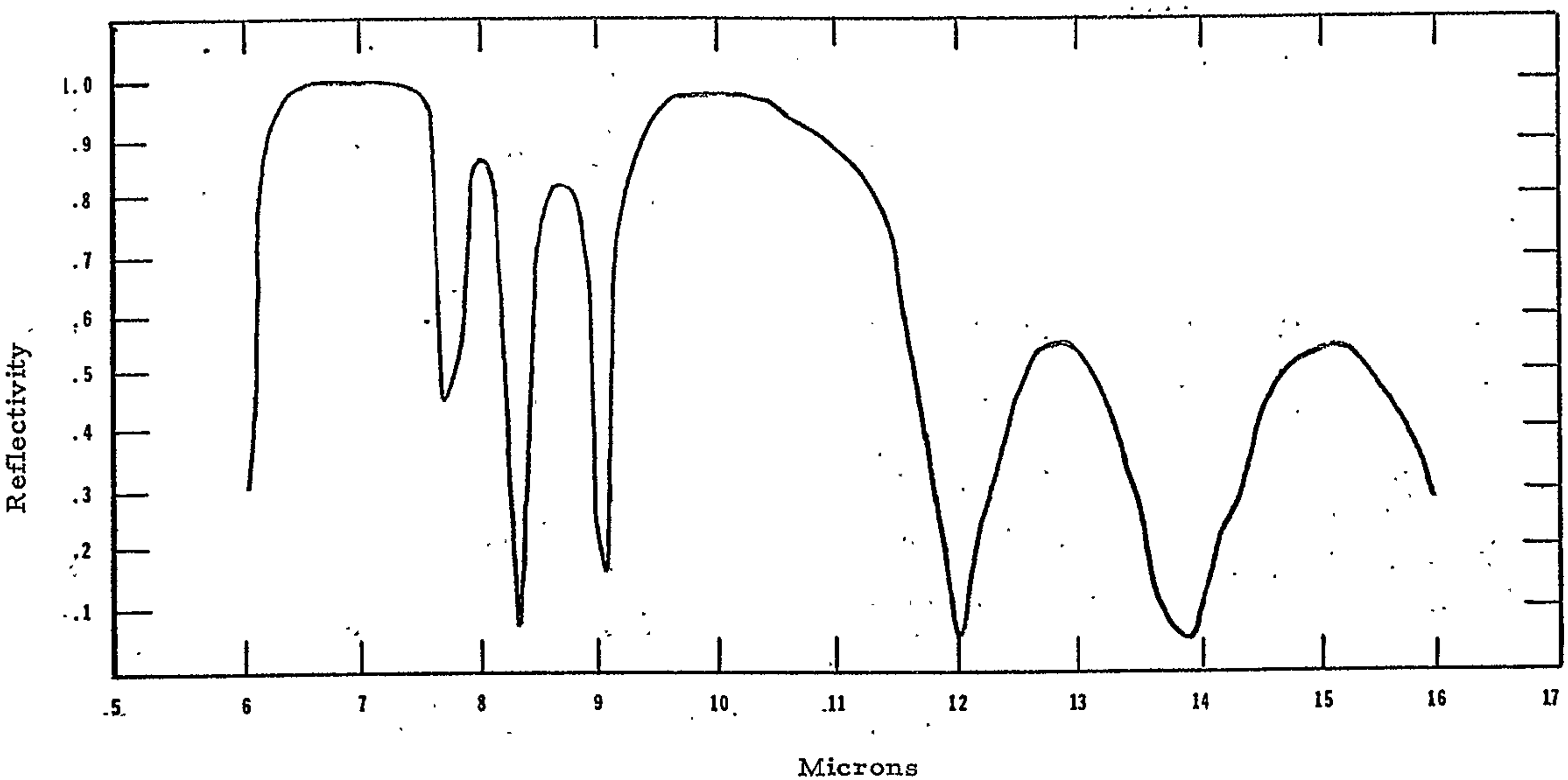


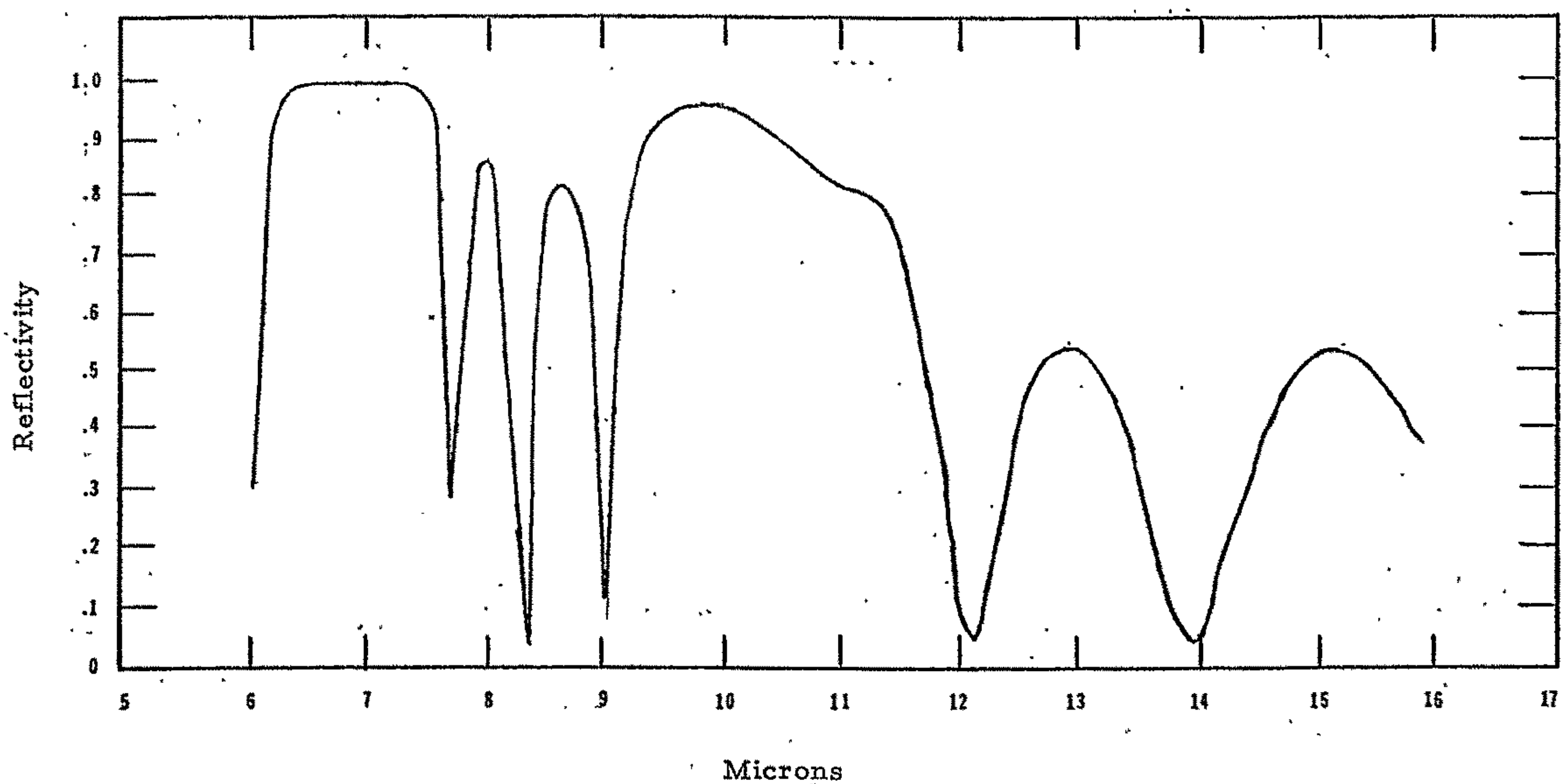
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
Reflectivity
4
5
6
7
8
9
10
11
12
13
14
15
16
Microns

FIGURE 4



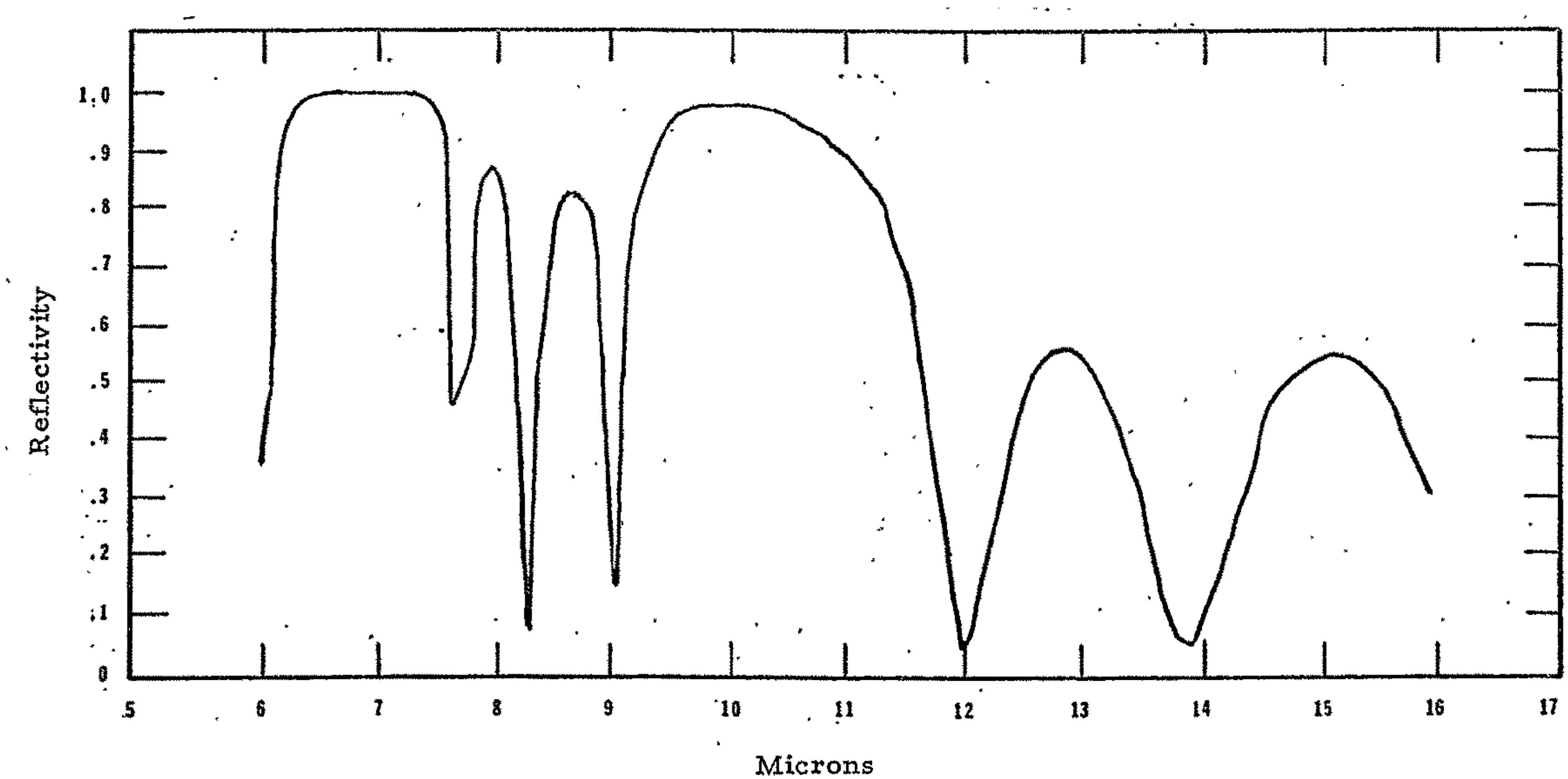
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
Reflectivity
5
6
7
8
9
10
11
12
13
14
15
16
17
Microns

FIGURE 5



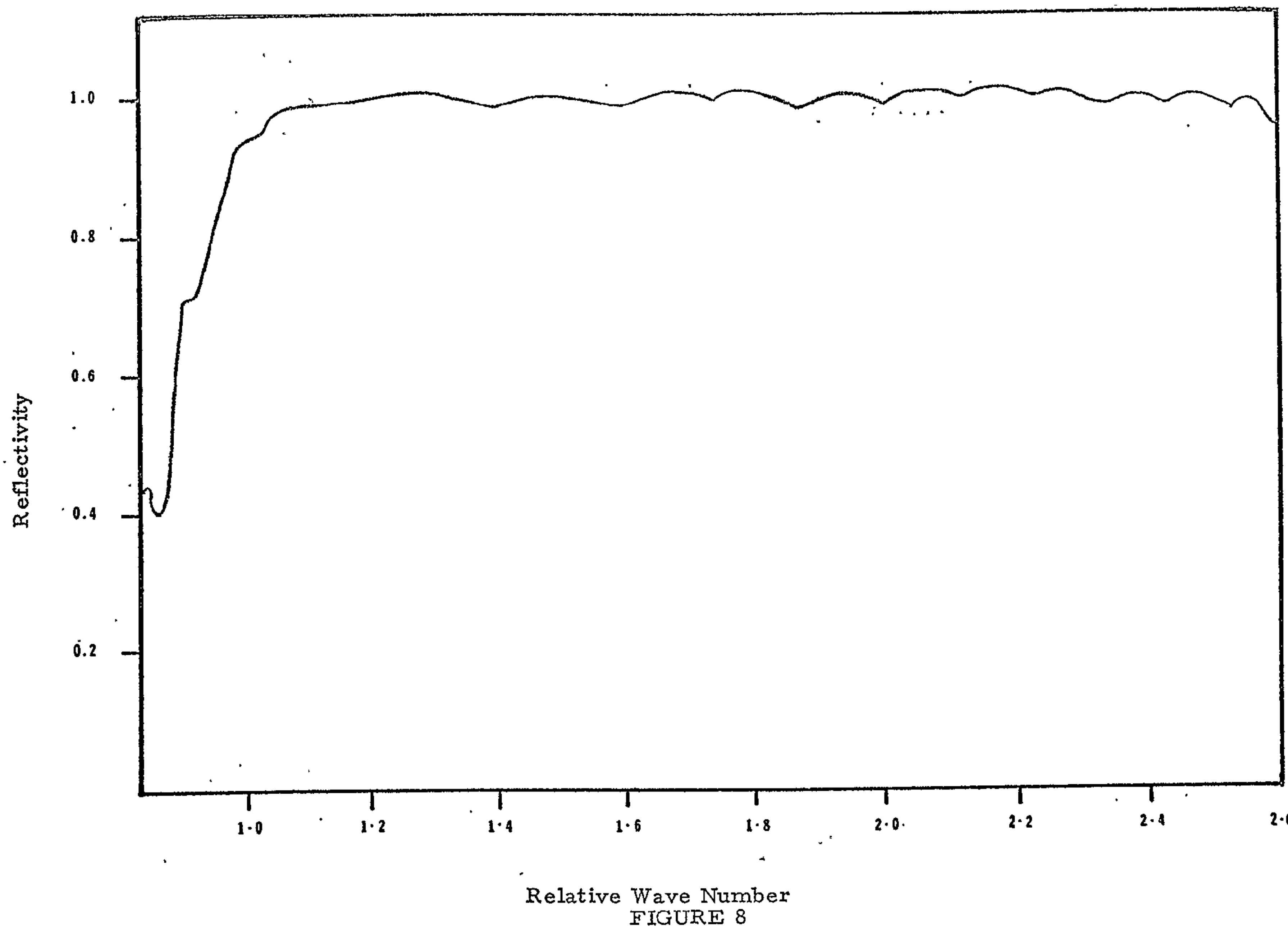
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
Reflectivity
5
6
7
8
9
10
11
12
13
14
15
16
17
Microns

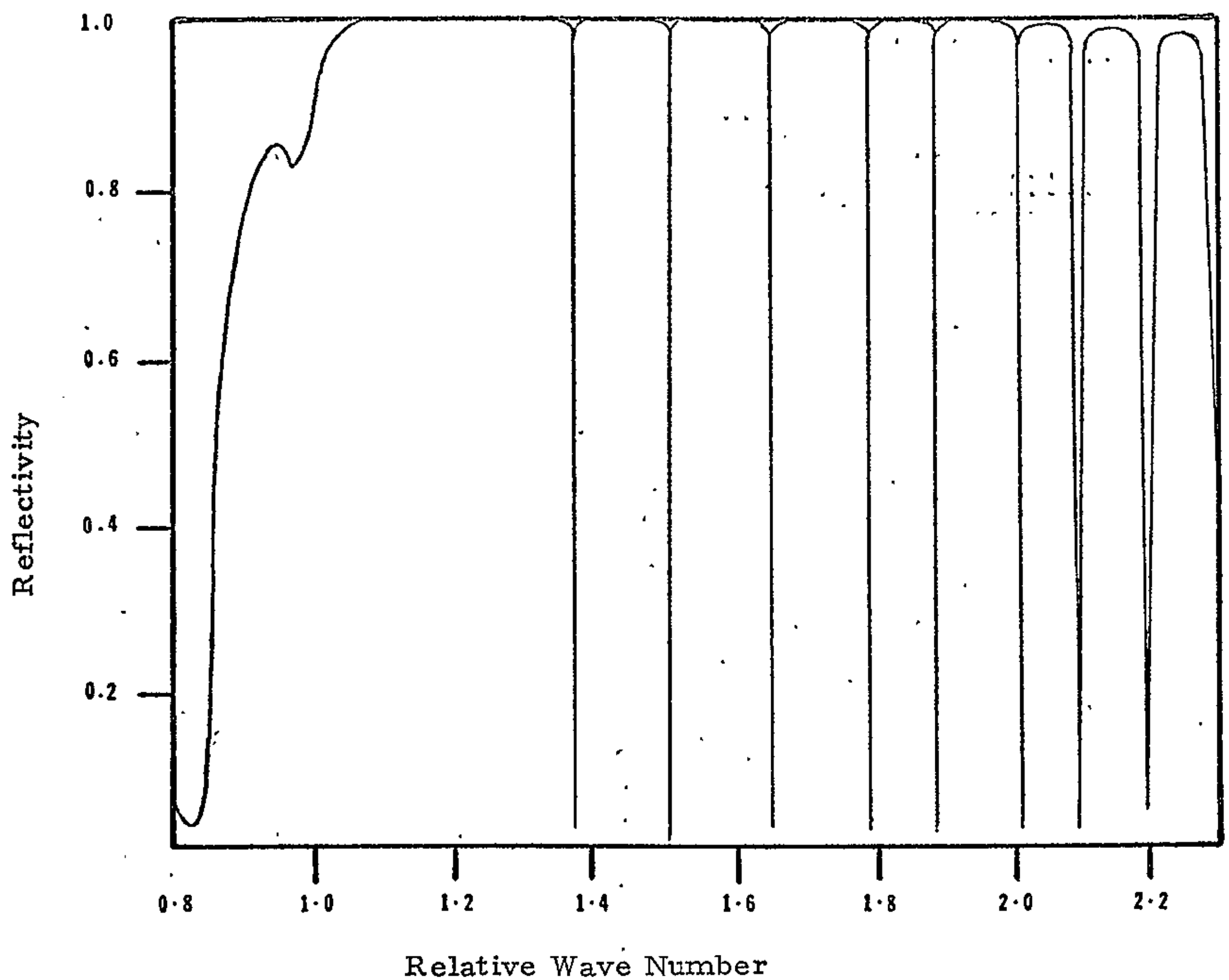
FIGURE 6



Reflectivity
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
5
6
7
8
9
10
11
12
13
14
15
16
17
Microns

FIGURE 7


1.0
0.8
0.6
0.4
0.2
Reflectivity
1·0
1·2
1·4
1·6
1·8
2·0
2·2
2·4
Relative Wave Number
FIGURE 8



1.0
0.8
0.6
0.4
0.2
Reflectivity
0·8
1·0
1·2
1·4
1·6
1·8
2·0
2·2
Relative Wave Number

FIGURE 9

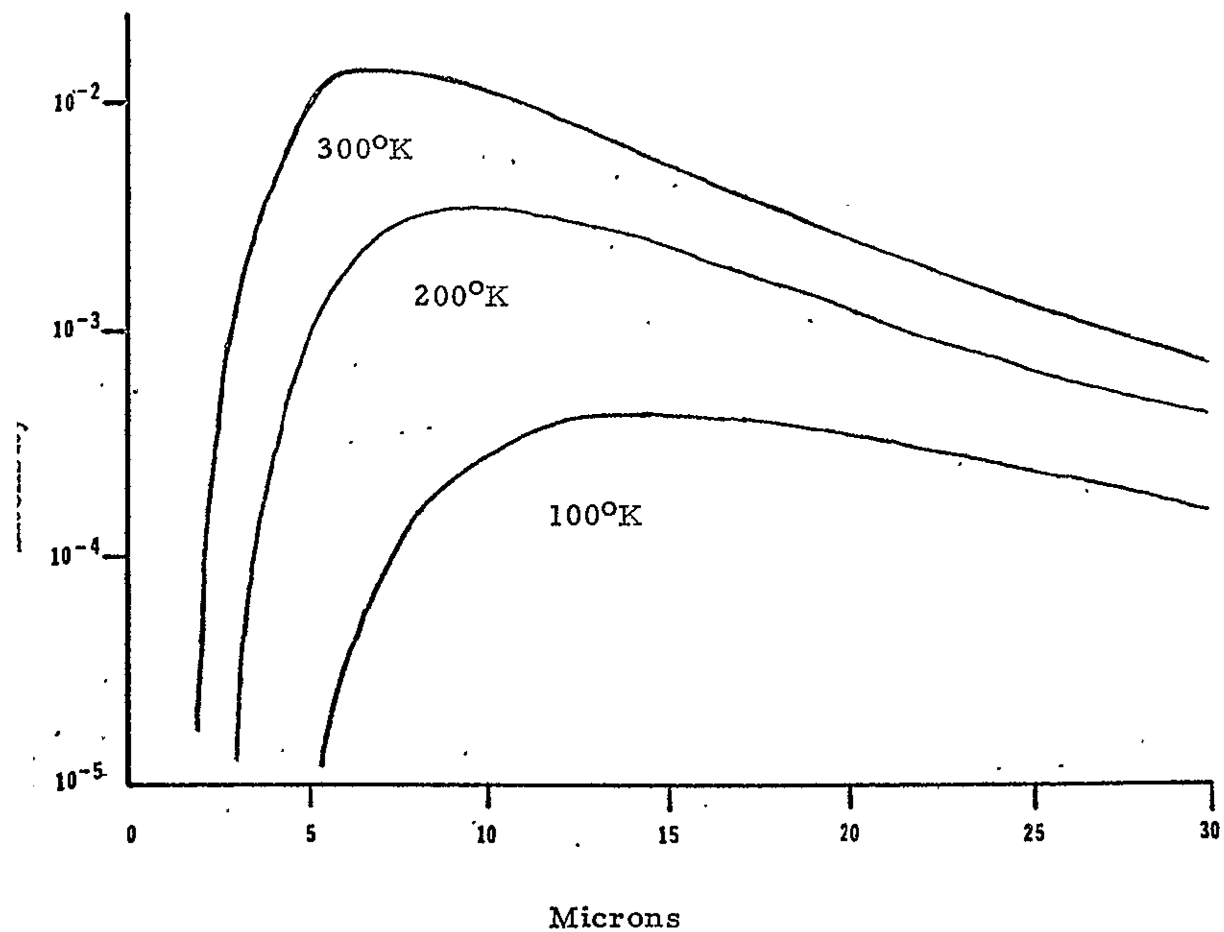
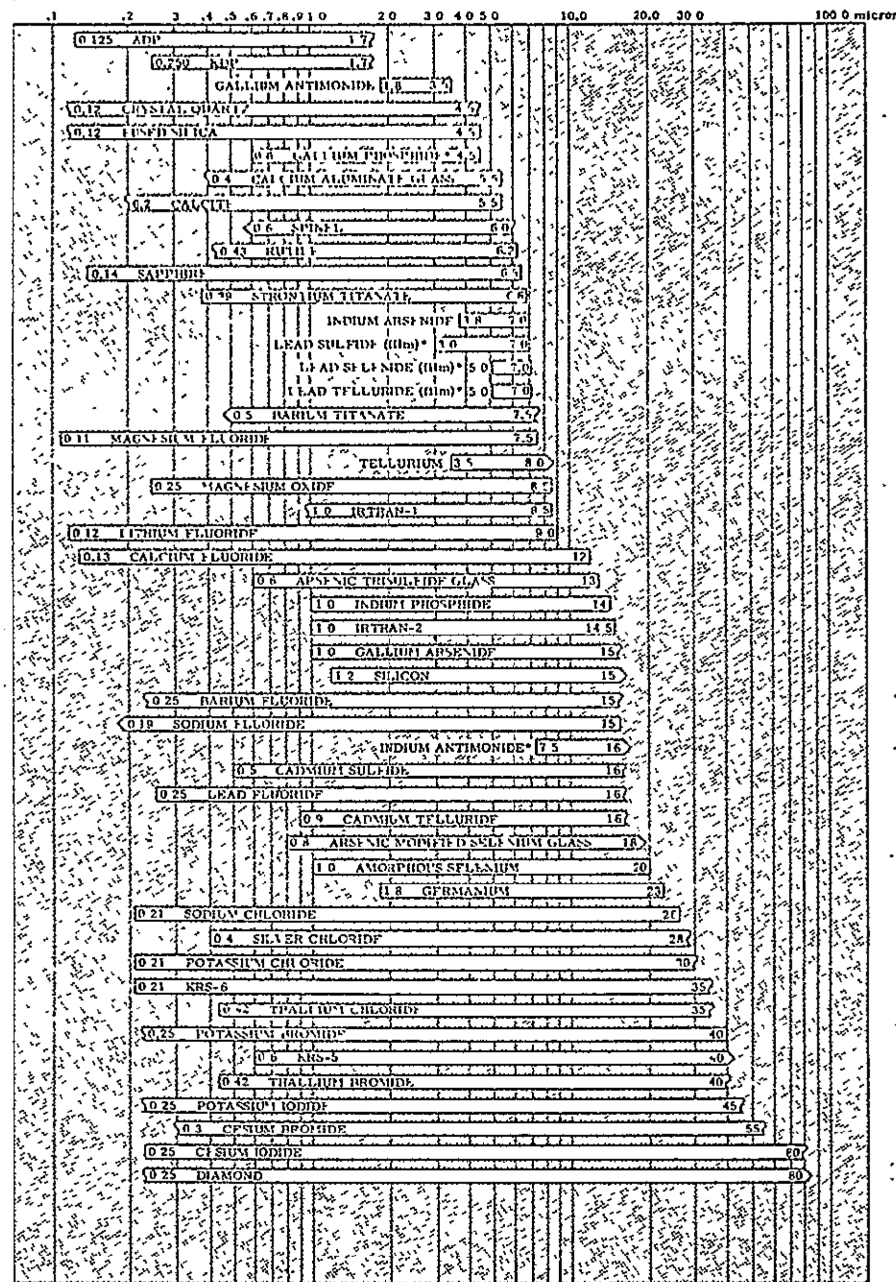
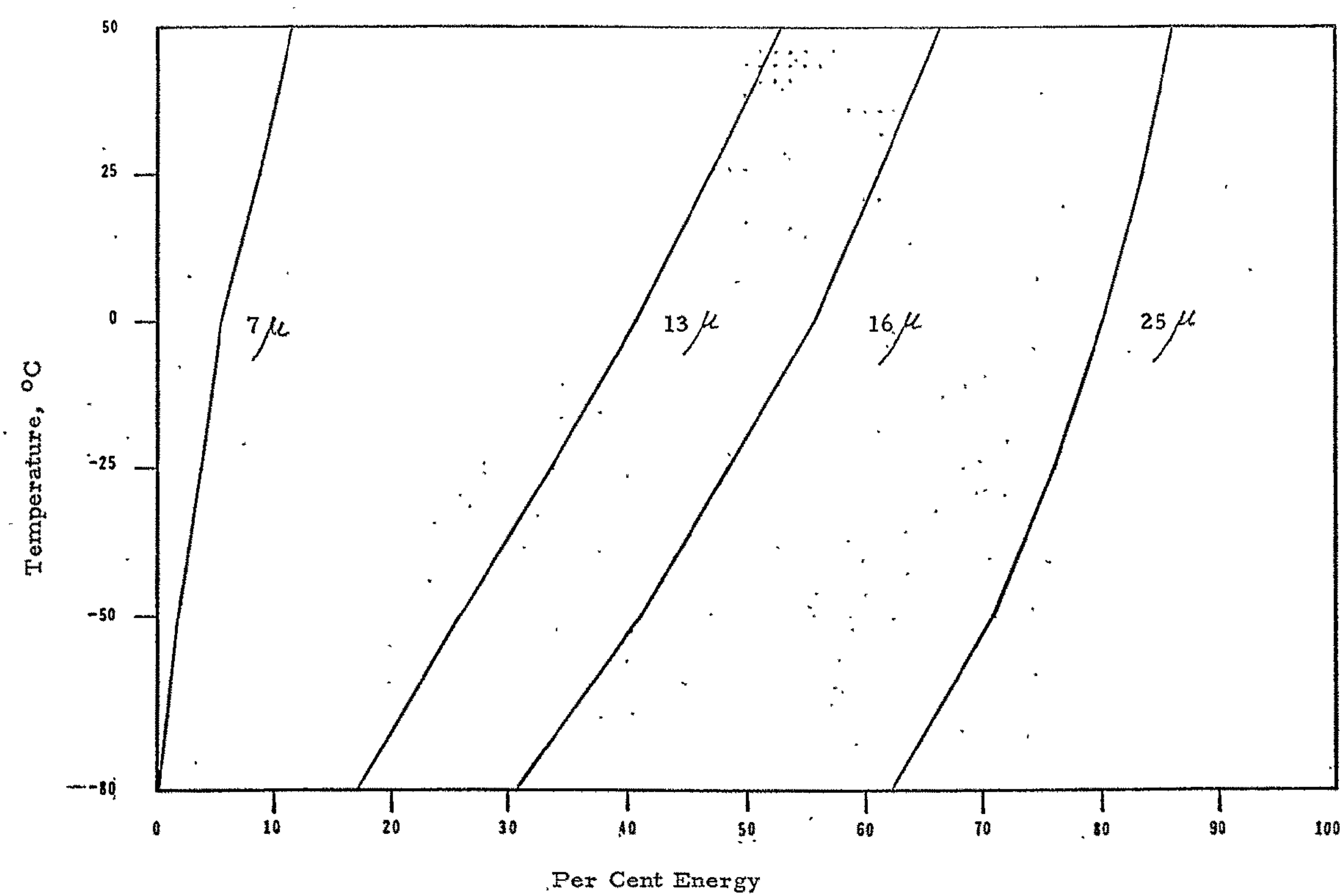


FIGURE 10



*Maximum external transmittance of less than 10%

FIGURE 11



50
25
0
-25
-50
-80
Temperature, °C
0
10
20
30
40
50
60
70
80
90
100
Per Cent Energy
7 μ
13 μ
16 μ
25 μ

FIGURE 12

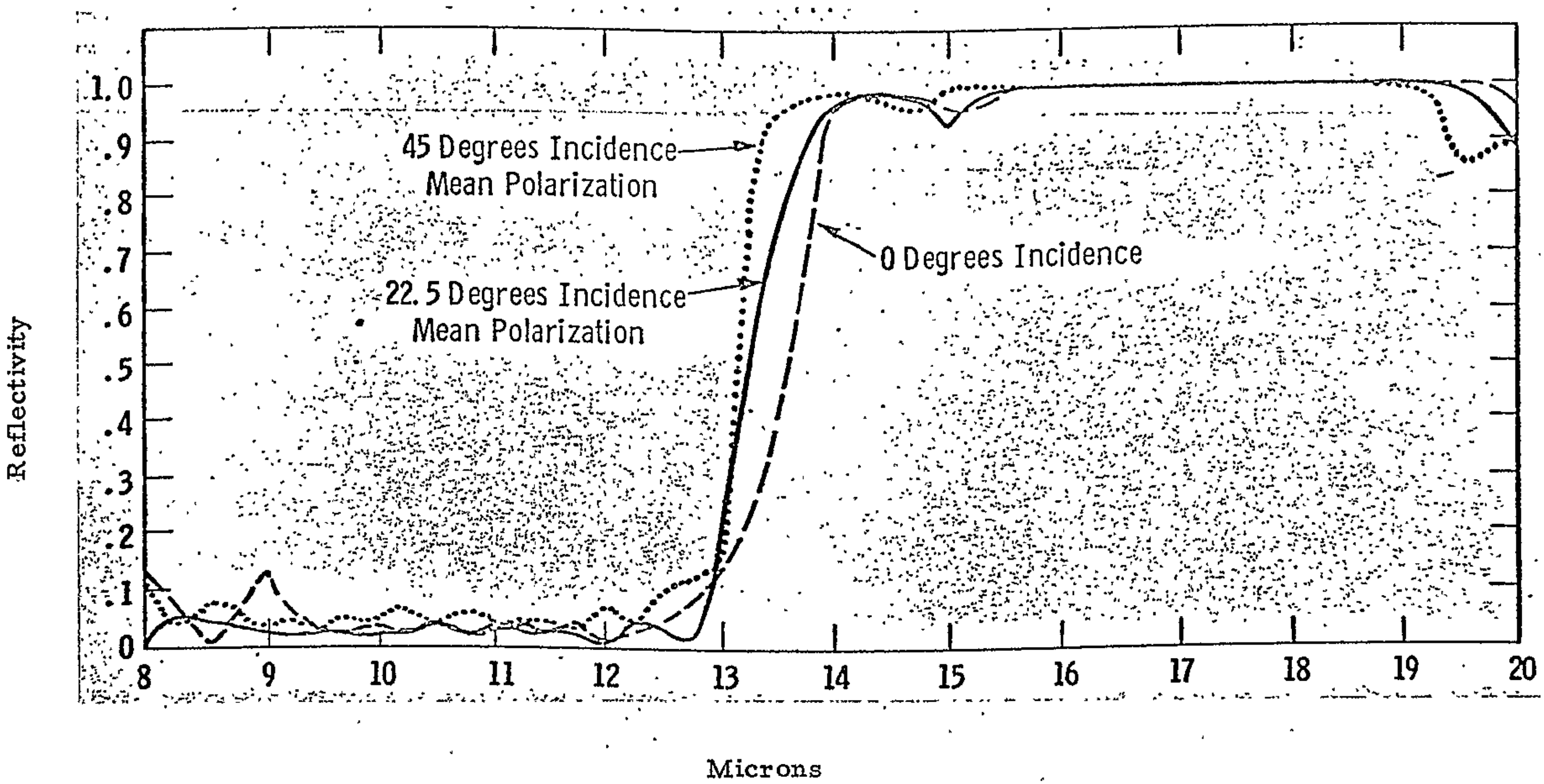

1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
8
9
10
11
12
13
14
15
16
17
18
19
20
Reflectivity
Microns
45 Degrees Incidence
Mean Polarization
22.5 Degrees Incidence
Mean Polarization
0 Degrees Incidence

FIGURE 13

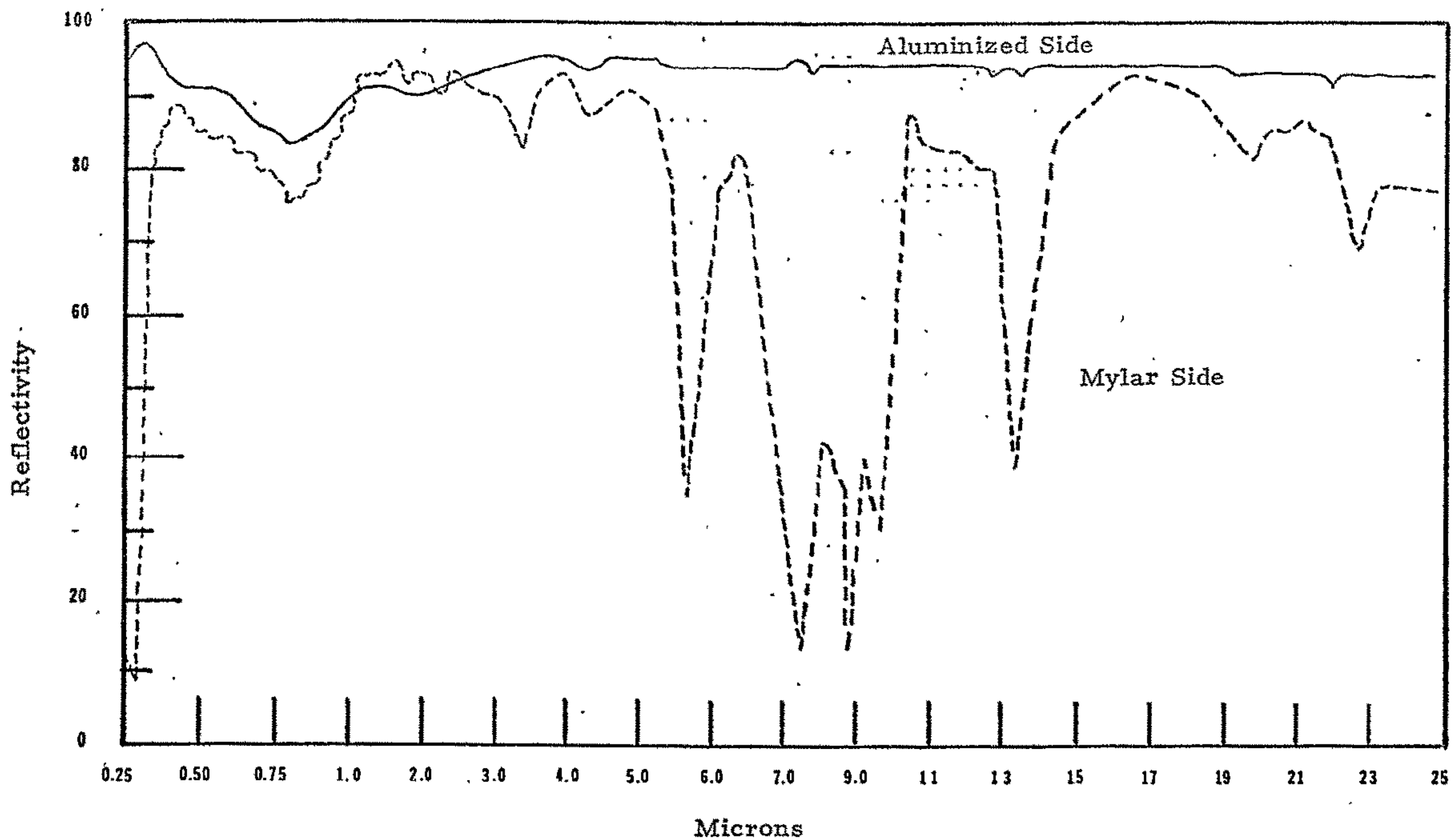

100
80
60
40
20
0
Reflectivity
0.25
0.50
0.75
1.0
2.0
3.0
4.0
5.0
6.0
7.0
9.0
11
13
15
17
19
21
23
25
Microns
Aluminized Side
Mylar Side

FIGURE 14

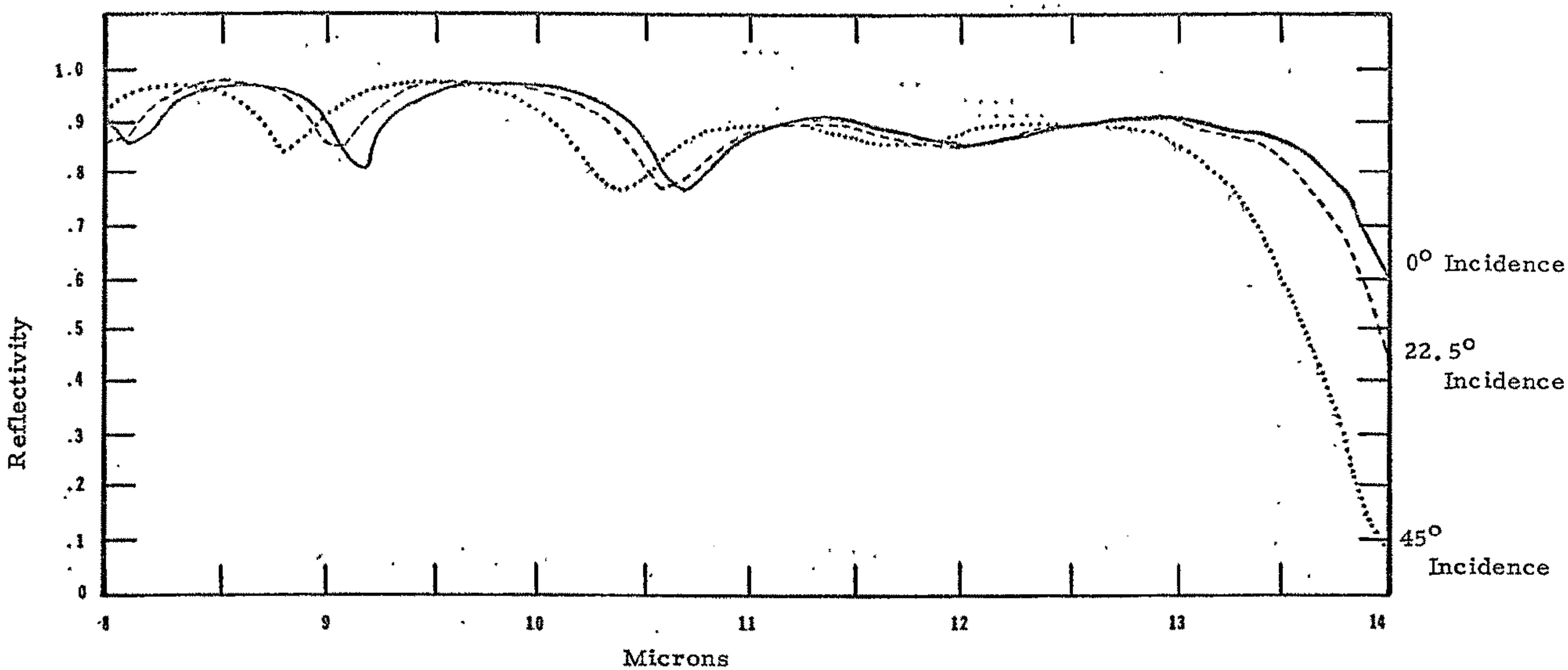

1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
Reflectivity
8
9
10
11
12
13
14
Microns
0° Incidence
22.5° Incidence
45° Incidence

FIGURE 15

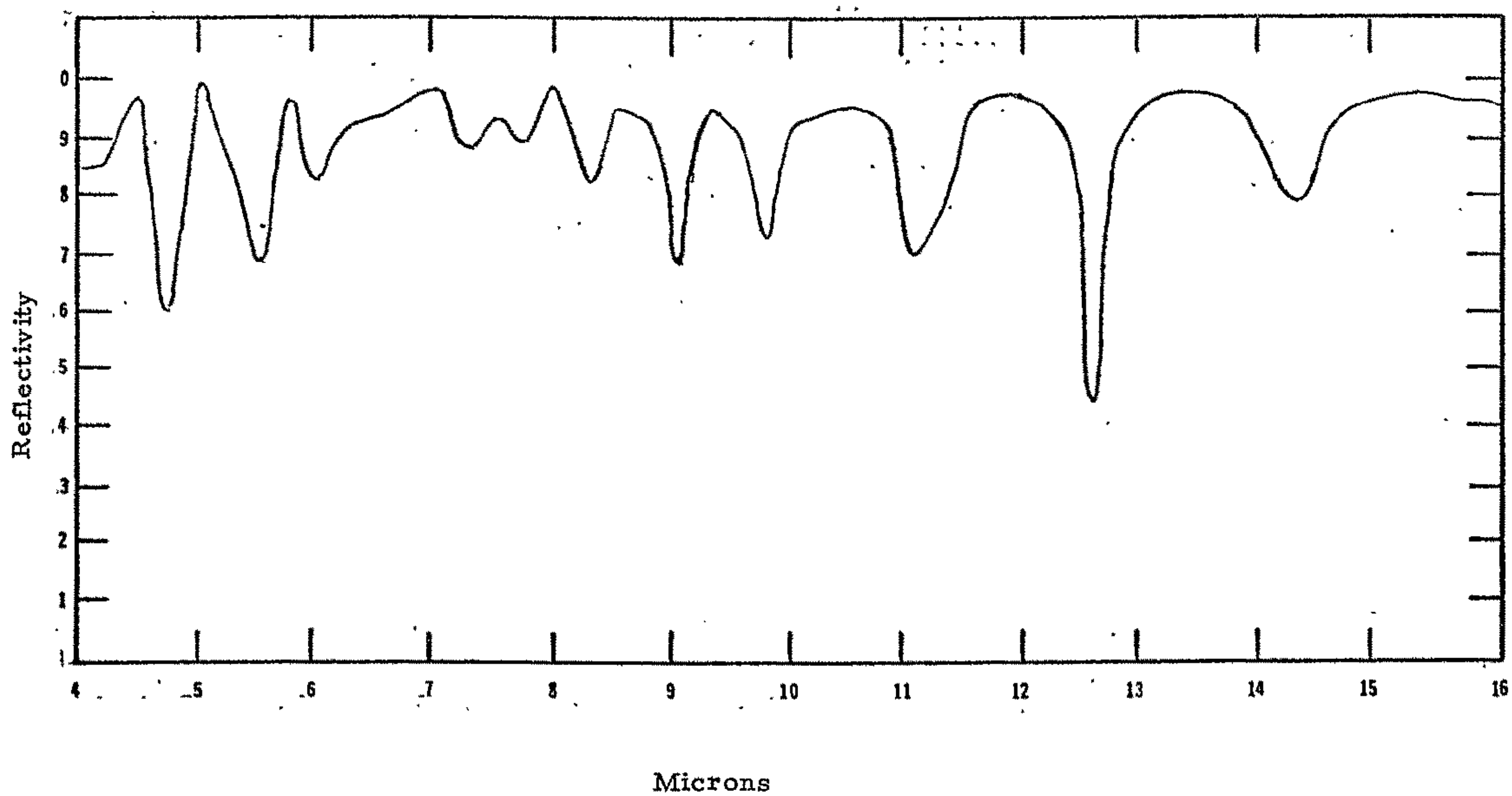

Reflectivity
0
9
8
7
.6
.5
.4
.3
2
1
1
4
.5
.6
.7
8
9
10
11
12
13
14
15
16
Microns

FIGURE 16

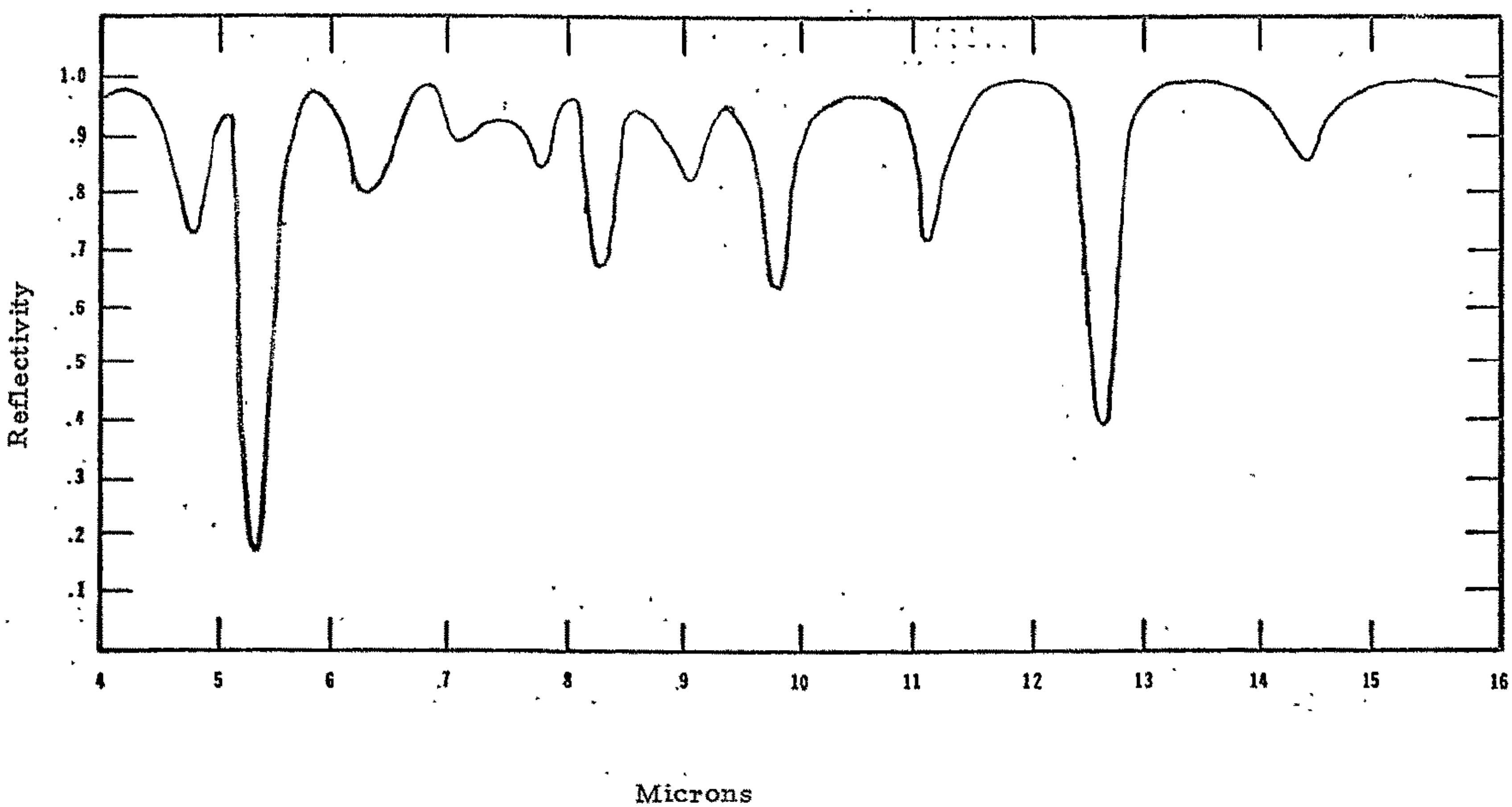
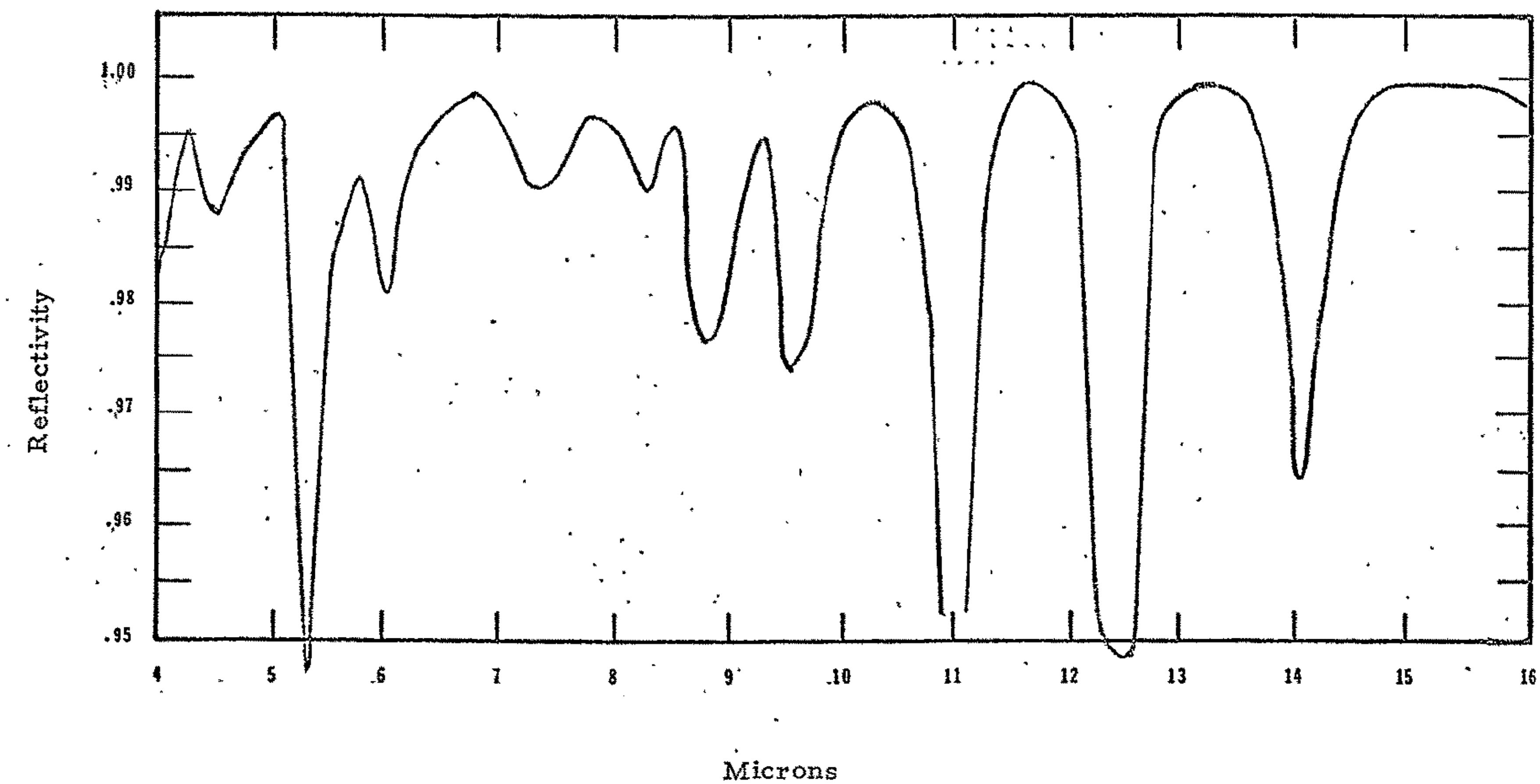

1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
Reflectivity
4
5
6
7
8
9
10
11
12
13
14
15
16
Microns

FIGURE 17


1.00
.99
.98
.97
.96
.95
Reflectivity
4
5
6
7
8
9
10
11
12
13
14
15
16
Microns
FIGURE 18

1.0 PRESENTLY ATTAINABLE LAMINAR CRYOGENIC THERMAL PROTECTION SYSTEM PERFORMANCE COMPARED TO ANTICIPATED FUTURE REQUIREMENTS

The most efficient cryogenic thermal protection systems are of the laminar "superinsulation" variety. These systems were discovered empirically in the late 1950's.

Since that time, no major conceptual changes in radiation shielding systems have evolved, although increasingly sophisticated engineering emphasis has been placed on proven but neglected system elements such as effluent vapor cooled shields and vessel supports, etc. The slow development of better materials and methods led to high performance space proven systems for storage of cryogenic fluids for long periods of time. However, current performance is lacking in comparison to performance which will soon be required. In a recent publication[1], engineers at the Manned Spacecraft Center have described some of the likely requirements of anticipated future space missions. Many of the near future cryogenic storage requirements for shuttle flight, orbiting labs, and lunar excursion are thought to be attainable at present although the development cost will probably be high. In stark contrast however, a near future Martian excursion seems definitely infeasible for the present due solely to cryogenic storage system requirements.

[1] M. L. Davis, R. K. Allgeier, T. G. Rogers, G. Rysavy, The Development of Cryogenic Storage Systems for Space Flight, NASA SP-247, U. S. Government Printing Office, Washington, D.C., 1970.

Far future missions appear to be eliminated from serious consideration at this time. One of the important conclusions of this report is that a factor of ten improvement in overall system performance would make feasible all future missions considered to date. It is axiomatic that factors of ten are not easily found in a technology such as this one. For an exact, complete picture of current external superinsulation systems technology, the reader is referred to earlier reports of this contract.[2]

In order to meet the requirements of some desirable near future missions, the only present alternative to improved laminar insulation systems is the addition of active cryogenic refrigeration systems. While this added system complication may be inevitable as the luxury of overboard gas venting is forgone to meet the rigorous demands of future space missions, refrigerators will add a further difficult complication to already complicated systems. If this addition becomes necessary, it will result in even greater emphasis on the need for better superinsulation systems.

1.1 Deficient Areas of Present Day Cryogenic Laminar Insulation Systems Technology

Granted that improved laminar insulation systems are desirable, one might reasonably ask how well understood are the limitations and shortcomings of present day systems. Surprisingly, the answer might come

[2] T. M. Flynn, External Insulations for Cryogenic Storage Systems, FINAL STUDY REPORT, Contract NAS9-10583, 1971.

back that while extensive empirical determinations of the properties of present systems have been carried out, and while the factors which degrade the performance of these systems have thereby been exposed, rarely have the basic physical mechanisms responsible for the system's function been considered in depth. Most attempts to improve system performance have settled on important, obvious, but somewhat peripheral matters, such as the reduction of conductive supportive heat leak (glass tension bands) or improved lamina evacuation properties ("Superfloc"), and these efforts have brought many present day systems to a level of performance thought unattainable previously. Indeed, a rough inspection of many current systems indicates that thermal energy reaching the pressure vessel flows about equally along conductive and radiative paths. Although there are many places where energy in either of the two paths can be interchanged from one path to the other, the separation of energy flow into these two basic transport mechanisms is now well justified by experiment[3].

In the conductive path, the mechanism of energy transport is well understood, on a phenomenological basis, and has been for years[4]. Although the conductive/strength properties of present day support system materials leave much to be desired, there has been no noteworthy deficiency in the full exploitation of these properties.

[3] R. H. Kropschot, private communication.

[4] H. S. Carslaw, J. C. Jaeger, Conduction of Heat in Solids, Oxford, 1959.

In the radiant energy channel, the situation is somewhat different. The calculation of energy transfer by this means in the complex geometries of laminar systems is not straightforward, and many simplifications have been introduced. (See Section 2.0) The result has been imperfect appreciation (if not imperfect understanding) of the properties and application of present materials. In addition, many practical deficiencies have appeared, and as a result, the entire matter is somewhat clouded. Although most cryogenic engineers, using empirical data, could design a functional superinsulation system, few have the understanding of the proper direction in which to proceed to improve radiation shielding performance. Since the theoretical aspect of this problem is considered in detail in the next section, it is appropriate to conclude this section by considering briefly some of the practical difficulties which plague radiation shielding technology.

Throughout all present systems, runs the implicit acceptance of the present concept of the laminar functional element.

The elementary functional element of superinsulation systems consists, in lowest terms, of a metallic reflector, separated from a series of others by a low conductivity spacer. While the spacers have grown quite sophisticated, the reflector is pure turn of the century technology. That is not to say that it is worthless or necessarily outmoded, but only that it should be examined carefully in the light of current requirements. The function of the reflectors, logically enough, is to redirect radiant energy incident on the system.

With the production of high vacuum in the early part of the century, the deposition of metals for reflection of electromagnetic radiation was achieved. The deposition of materials in vacuum is even today fraught with pitfalls for the unwary, and the deposition of metallic coatings is no exception, long though the history may be. The practical difficulties which cryogenic engineers encounter often center around poor adhesion of the metal coating and spacer, or thin spots in the metallizing. For example, there is a well documented instance of coating destruction by accidental admission of water vapor to a cryogenic superinsulation system.[5] Additionally, since it is well known that high infrared reflectance is not necessarily related to high visible reflectance (coatings have been deliberately produced with low visible and high infrared reflectance[6]), many engineers are reluctant to reject obviously poor coatings, for fear of being overruled by an emissometer measurement.

If this were not enough, a worse problem appears when the radiation shielding must be penetrated by fluid supply piping. The pipe compromises the radiation shield performance severely by reducing the local thermal

[5] T.M. Flynn, Ibid.

[6] G. Hass, H.H. Schroeder, A.F. Turner, Mirror Coatings for Low Visible and High Infrared Reflectance, J.O.S.A. 46, 31, (Jan. 1956).

gradient through the shield, and often by the creation of random small direct radiation windows to the tank. Additionally, the awkward geometry of the tank/piping juncture makes it difficult to insulate the penetration reproducibly from installation to installation. Finally, unless carefully executed, the penetration insulation design can compromise the entire radiation shield performance, as excess heat carried in along the piping can be transported throughout the interior of the lamina by the high lateral thermal conductivity of metallized film. Frequently this factor is one of the most uncontrollable variables in the construction of experimental systems. It undoubtedly restricts severely the effectiveness of penetration design approaches.

In spite of these difficulties, metals are used exclusively for superinsulation reflectors solely because they have high reflectance throughout the infrared (cf Fig. 1).

2.0 PHYSICAL PROCESSES ASSOCIATED WITH THE FUNCTION OF CRYOGENIC THERMAL PROTECTION SYSTEMS

The effort to improve the function of the reflecting element of these systems has centered on an elusive property of the material denoted as its emissivity. The property is elusive for reasons well known to thermal control designers. It is difficult to measure accurately and under controlled conditions, and changes rapidly, unpredictably, and frequently inexplicably.

To put all this in proper perspective, it is well to consider briefly what is known about the basic physical processes which influence the thermal properties of solids and the transfer of radiant energy in laminar thermal protection systems. It is also instructive to consider briefly how knowledge of these basic processes is translated into predictions of engineering performance, and what success has been accumulated by these efforts.

2.1 Thermal Radiation Properties of Solids

Thermal radiation, by definition[7], is that electromagnetic radiation produced solely as a consequence of the thermal energy of materials. The thermal energy of objects is contained in the motions of their constituent atoms and molecules. These motions, experienced as heat, also cause the

7 E.M. Sparrow, R.D. Cess, Radiation Heat Transfer, Revised Edition, p. 4, Brooks/Cole Pub. Co., Belmont, Ca. 1970.

emission of radiation. The absorption of radiation can likewise produce heat, but the radiation itself is not heat energy. Infrared radiation in particular, produces heat readily because many substances absorb it strongly as a consequence of their molecular structure. Many substances have broad regions of absorption for infrared energy and consequently exhibit rapid heating in the presence of broad spectrum infrared radiation. In addition, some substances show very strong absorption for specific frequencies. Cryogenic liquefied gases exhibit the latter behavior.

Radiant energy absorbed by atoms and molecules can be dissipated by many processes, one of which is subsequent remission at the same or at a longer wavelength. While this is familiar to everyone in the case of objects such as black bodies, the phenomena are quite complex, and the branch of physics known as quantum mechanics was developed specifically to explain them.

Solid objects located in a room ambient (300°K) thermal environment have a considerable amount of thermal energy, and emit to and absorb from their surroundings an unceasing stream of electromagnetic energy in the 4 - 35 μ spectral region, with energy peaked aroung 10 μ . A phenomenological description has been developed to characterize the behavior of objects in this regard. Thermodynamically, a black body is the most efficient uncontrolled radiant source which can be devised. It is natural to measure other objects according to the black body standard, and that measurement leads to the property designated as emissivity for specularly reflecting materials (or emittance for diffusely radiating materials). The total hemispherical emittance of a material is defined to be the power emitted by the object at a given temperature per unit time and area at all wavelengths into the hemispherical space exterior to its surface, ratioed to the same quantity for a black body at the same temperature. Hence:

$$\epsilon = \frac{\int_0^\infty \epsilon_{\lambda T}\, e_{b\lambda T}\, d\lambda}{\int_0^\infty e_{b\lambda T}\, d\lambda}$$

where $\epsilon_{\lambda T}$ is the monochromatic emittance of the object at temperature T, and $e_{b\lambda T}$ is the monochromatic emittance of the corresponding black body.

While this seems quite straightforward, in practice the emittance of any common material does not generally represent the intrinsic properties of the material, but rather the condition and properties of its surface. It is this fact which makes insulation materials so difficult to deal with. Furthermore, seldom if ever has the monochromatic emittance been measured for insulation materials, although that circumstance may change slowly. The measurement is difficult to make, but is important to accurately characterize many practical engineering problems.

Another parameter important to discussions of the properties of radiation shields is the total hemispherical absorptance α. In contrast to the emittance, it is not defined by reference to a black body, but rather by reference to the spectral distribution of hemispherically incoming radiation per unit time and area, denoted H_λ:

$$\alpha = \frac{\int_0^\infty \alpha_{\lambda T} H_\lambda \, d\lambda}{\int_0^\infty H_\lambda \, d\lambda}$$

From Kirchoff's law[8] which rests directly on the second law of thermodynamics,

$$\epsilon_{\lambda T} = \alpha_{\lambda T}$$

In general however, because of the variation of H_λ $\epsilon \neq \alpha$. For several particular circumstances ϵ and α can be related, or the assumption of equality made.[9]

The commonest circumstance is the assumption that $\epsilon_{\lambda T}$ and $\alpha_{\lambda T}$ are not functions of wavelength. Such objects by convention are called gray bodies. While in the first order, many materials behave in this fashion,

[8] M. Planck, The Theory of Heat Radiation, Dover Publications, New York, 1959.

[9] E. M. Sparrow, R. D. Cess, Ibid, p. 41.

metals in particular have rapidly changing monochromatic emittances in the infrared[10]. In spite of this, as we shall see later, this is the assumption commonly made in laminar insulation radiant energy transfer calculations.

In the only other circumstance of interest here, radiation from a black or gray body, temperature T_i, incident on a metallic surface, temperature T_s, yields an absorptance

$$\alpha \doteq \epsilon(\bar{T}) \ ; \quad \bar{T} = \sqrt{T_i T_s}$$

The foregoing shows that, since the spectral content of radiant energy will be shifted to longer wavelengths at each surface encountered as it progresses into the laminar radiation shield system, and because of the region of the spectrum involved, the assumption $\alpha \doteq \epsilon$ is unjustified, and the black or gray body approximation is probably invalid.

As if this were not enough, the thermal radiation properties of metallized film probably must be considered in a more complex fashion than the above would indicate. This is unfortunate if true, for much engineering treatment of this material is couched solely in the terminology described above, and is inadequate, inaccurate, or oversimplified to the point of little meaning.

[10]E. M. Sparrow, R. D. Cess, Ibid, p. 64

In actuality, metallized plastic film represents a material system whose radiant energy transfer properties have more roots in optics than in the thermal methods developed to characterize the engineering properties of diffusely reflecting bulk materials. Even the characterization of those materials is difficult, and because of the extreme dependence on surface properties, one really deals with the properties of films in those cases whether it is explicitly recognized or not.

Metal coatings on plastic film have dimensions of considerably smaller size (roughly $\lambda/200$ or less) than the radiation wavelengths involved in the energy transfer. The surface formed by the metal properly deposited has decidedly optical properties; it is smooth, and exhibits specular reflection. Its absorptive properties are a strong function of thickness at a given wavelength[11]. Additionally, the reflectance is sensitive to the coating thickness. This is illustrated by Figure 2 , which shows, for commonly accepted optical constants, the reflectance of aluminum versus thickness in the visible. The optical constants of metals are <u>not</u> well known in the infrared, and are somewhat in doubt even in the visible. For optical materials, the connection between optical constants and the monochromatic and total emissivities are demonstrated by Sparrow and Cess[12].

[11] O. S. Heavens, <u>Optical Properties of Thin Solid Films</u>, p. 202, Dover Publications, New York, 1965.

[12] E. M. Sparrow, R. D. Cess, Ibid, p. 63ff.

All of these factors complicate the calculation of radiant energy transfer in such systems, and the statement of emittance for the material conveys relatively little information. Probably a better measure of film radiation transfer properties is the reflectance (also difficult to measure properly). However, the emittance of materials in bulk is likely to be entirely a different matter from the emissivity of materials in film form. Because of wave interference effects in film assemblies, the absorptance in the film can be an entirely variable matter, depending on the case. Nor should bulk emittances provide much insight to the thermal emission properties of films, as they differ greatly in microstructure. The problem is further compounded by the great variability of bulk emittance measurements.

As guide for what one can expect for dielectric films, one can consider what is known of the absorptive part of the refractive index in the 2 - 20 μ range of the infrared. For aluminum, there are <u>no</u> consistent measurements in this region[12], but order of magnitude, the extinction coefficient K (which is related to the depth of penetration and attenuation of the wave into the surface) is approximately 50. For germanium, in contrast, K is roughly 0.1 in this region, a factor of 500 smaller. Hence one would expect much less absorption of incident energy using germanium than aluminum.

[12] A. J. Moses, <u>Refractive Index of Optical Materials in the Infrared Region,</u> Electronic Properties Information Center, Report DS-166 (Jan. 1970).

In practice, as noted earlier, there is a lack of information, except for experience with dielectric filters in the visible and near infrared. The absorptance of these devices is normally lumped with the scattering from defects in the films, and is usually so low as to be measurable only with highly sensitive equipment. The values ordinarily expected for absorptance are given in Section 3.4. Consequently, we expect the intrinsic thermal emissive properties of film assemblies to be quite good if the correct materials are used for the spectral region involved, and if the device is properly designed. One thing is certain: reflected energy is not transmitted or absorbed, and the worst case emittance can be no worse than the total unreflected energy. Energy not reflected enters the system at any rate, and the only virtue of low emittance is that the degradation of the spectral content beyond the wavelengths where it can be easily controlled (reflected) proceeds at a slower rate.

Finally, let us discuss the thermal radiation properties of nonconductors briefly in order to highlight further the paradoxes which the emittance characterization produces.

It will be agreed by anyone asked that electrically nonconducting media have very high emittance compared to metals, ranging from 10 to 100 times greater. This is due to the fact that such materials become opaque for the most part at infrared radiation wavelengths. However in the visible, for example, many of these materials such as glass, have a purely real refractive index and hence do not absorb that radiation. There are no materials with this property in the infrared, but there are many which have useful transmission, and do not absorb nearly as strongly as the metals, due to their shortage of free electrons. Although emittances for these materials are not apparently published in the open literature, there is little reason to believe they would be as low as those of metals, based on bulk emittances. We have stated earlier that candidate materials selected for dielectric reflectors are expected to exhibit low emissivity when assembled into a device. This will produce a conflict reaction from many persons when dielectrics are mentioned.

2.2 Radiant Energy Transfer Through Lamina

The standard methods of calculating the transfer of radiant energy between diffuse surfaces are summarized in an article by Sparrow[13]. Since super-insulation radiation shields have specular properties, the above methods do not really give an accurate picture of radiation transfer in that case.

[13] E. M. Sparrow, Modern Developments in Heat Transfer, Ibele, ed. Academic Press, New York, 1963.

Traditionally, very simple methods have been used to estimate the heat transfer in insulation systems. The simplest of these is the so called floating shield equation[14]. This equation, quoted widely, is used for order of magnitude calculations, and does not agree with observations to within a much smaller margin than that.

The derivation is straightforward; the net radiant heat flux between the i th and the i th + 1 surfaces of N floating radiation shields is

$$q_{i,i+1} = \frac{\epsilon}{2} \sigma \left(T_i^4 - T_{i+1}^4 \right)$$

There are N + 1 sets of these equations. The sum of the left hand terms is (N + 1) q where q is the total flux. The intermediate terms of the right hand side cancel, so that

$$q = \frac{\epsilon}{2(N+1)} \sigma \left(T_H^4 - T_C^4 \right)$$

T_H and T_C are the temperatures of the exterior surfaces bounding the shields.

[14] R. P. Caren, Proceedings of Cryogenic Technology Symposia of Cryo-68, Tinnon-Brown Inc., L. A., Ca., 1969.

To properly calculate the complete heat transfer (in a metallized film) due to all sources, the conductive effects must also be included. By normal calculational procedures, this amounts to making the problem seriously nonlinear, since one must solve the equation of radiative heat transfer simultaneously with the conduction equation. Generally speaking such efforts have also had little success[15].

The most promising approach to the problem, in view of the optical nature of many aspects of the process, is the Monte Carlo method. Using this procedure, a computer simulates the system, and tabulates the consequences of the simulation ground rules. This seems quite promising, considering the great success which the technique has yielded with difficult problems of a similar nature in the past. For example, reflections of radiation in laminar systems give rise to polarization effects in the transmitted and absorbed spectrum. The method can easily take this into account. The coupling of radiation and conduction processes also presents no problem.

This evaluation procedure would yield a great deal of understanding of laminar insulation systems which probably cannot be gotten any other way. In order to press hard on insulation improvement, it needs urgently to be done.

[15] R. P. Caren, Ibid.

2.3 Secondary Conductive Energy Transfer Effects

It is important to note here that a radiation shield material which did not have the high lateral thermal conductivity associated with present materials would also improve the conductive thermal leak to the pressure vessel. This is a systems - related benefit which might not be expected from efforts to improve only the radiation shield system component.

In practice, with proper attention to the method of improvement attempted this might prove to be a significant effect.

Little quantitative data on this effect in present systems has appeared in the literature. Usually the effect is quite dependent on system geometry, seam treatment, etc. For the proposed new material, an indication of the lateral conductivity would most reliably be obtained from samples. If the same test were performed on metallized film, a system independent comparison of these materials could be obtained.

2.4 Thermal Radiation Properties of Cryogenic Liquefied Gases

In the past few weeks, results of recent basic scientific research have been discovered bearing directly on long term cryogenic storage. In two works (one as yet unpublished), M. C. Jones of NBS, Boulder Laboratories

Cryogenic Division has summarized and extended what is known of the infrared absorption properties of liquid cryogens. The results are startling.[33,34]

In the gas phase, the homonuclear diatomic gases H_2, N_2, etc. exhibit practically nonexistent infrared absorption. CO and O_2 deviate slightly from this behavior, the former having a permanent electric and the latter a small permanent magnetic dipole moment. In compressed states, selective infrared absorption first appears, and is quite pronounced in the liquid phase of each gas.

The observed absorption is quite selective. Let us consider LH_2, LO_2, and LN_2.

In LH_2, the absorption occurs in three infrared regions. There are three absorption peaks which lie in the 2 - 3 μ region, called the fundamental absorption band. A second band, centered at 25 μ is called the translational band, is broadly centered around 100 μ. The first two bands have their origin in collisional induced interactions which allow the molecule to change its vibrational and rotational states, absorption process whereby two molecules translational band arises from an absorption process whereby two molecules in collision change their kinetic energy by photon absorption, without changing their internal states. The same general types of processes have

[33] M. C. Jones, NBS Technical Note 390, April 1970.

[34] M. C. Jones, to be published.

been observed in all of these liquefied gases. The rotational band shows the greatest strength in hydrogen, having a peak absorption coefficient of 3.5 per cm., about 20 times the peak strength of the translational band. The strength of the rotational band is also greater for parahydrogen than for normal hydrogen, by a factor greater than 3.

Jones has calculated from these data that 300°K black body ratiation is 63% absorbed in 1.9 cm of parahydrogen, while 2.5 cm is required for the same absorption in normal hydrogen. The fundamental band, he observes, accounts for at most 3×10^{-6} of the absorption in this case, and can be neglected.

LO_2 has four observed infrared absorption bands. A series of bands called the atmospheric bands are produced by electronic transitions. They lie above 2 μ, and can be disregarded.

The fundamental band of oxygen is centered at 6.4 μ, and an overtone band, 100 times less intense, occurs at 3.2 μ. The rotation band of oxygen centers at 100 μ. The importance of these bands in providing energy absorption within the fluid varies with temperature, the fundamental dominating for high and the rotational band for low. The overtone band is not important. The translational band is not distinguishable separately for oxygen, overlapping the rotational band.

For liquid nitrogen, the rotational band is centered around 100 μ and has a strength roughly equivalent to that for normal hydrogen, and nearly 3 times stronger than the same band in oxygen. We have been unable to discover measurements made for higher frequencies in nitrogen, and must therefore abandon a discussion of its properties for the present.

These results indicate that it is absolutely imperative to restrict the passage of certain infrared frequencies into liquefied gas. The optical concept meets that criterion better than any other approach at present. In addition, it is imperative to prevent absorption and remission of energy at a subsequently critical wavelength, which we believe is also satisfied by the optical material. It is conceivable that this goal can also be met by the use of superconducting radiation shields, but there are some limitations to that approach as well. In short, an optical approach to cryogenic insulation seems well justified by the evidence gathered at this point.

3.0 A NEW APPROACH TO THE DESIGN AND PRODUCTION OF CRYOGENIC INSULATION MATERIAL

An approach for sidestepping some of these problems has appeared as a consequence of the resurgence in optics experienced in the last few years.

Dielectric materials are anathema to cryogenic engineers because of their high emissivity in bulk. The properties of the bulk material are determined by the physical structure, which includes a scarcity of free conduction electrons, rigid energy conditions imposed on the electrons which are available for conductivity, etc. However, because of these properties, the thermal conductivity of dielectrics is orders of magnitude lower than that of metals. By making use of the phenomenon of interference it has proven possible to manufacture all dielectric structures with high reflectance and low absorbance. These remarkable systems are manufactured by multilayer coating technology, and are responsible for the existence of lasers. Most lasers are visible devices, however, and the extension of this technology to wavelengths of interest in cryogenic systems has not been carried out. It is the purpose of the discussion from this point to examine the feasibility of this extension.

The purpose of a cryogenic thermal protection system can be more clearly understood if one considers the objective and why it is not achieved fully. The goal is to prevent energy, predominantly thermal, from entering a pressure vessel containing a liquefied gas. The vessel is contained within the structure of a spacecraft, and that structure is the source of unwanted energy, provided the vessel is properly shielded (by the structure) from direct solar or planetary radiant energy.

Since the temperature of deep space is approximately 3°K except for these sources, deep space is of no concern to the discussion as an energy source. The operating temperatures of spacecraft structures vary widely, as a matter of design. For materials reasons however, the normal range of temperatures encountered typically ranges from 300°K to 180°K. If the spacecraft were considered as a black body, this implies distributions of radiant energy in accord with the well known black body curves (Fig. 10). In the case of the higher temperature, this implies an energy distribution substantially beginning between 3 and 4 μ, peaking at 9.5 μ, and having 80 per cent of its energy at wavelengths shorter than 22.5 μ. For the lower temperature the corresponding numbers are 5 - 6 μ, 16 μ, and $\sim$ 35 μ. Generally, the range of wavelengths from which tanks must be shielded is thought to lie from 4 to 400 μ[16]. Because of the difficulty associated in the production and measurement of far infrared radiation, measurements beyond $\sim$ 30 μ are generally not attempted, and metallic films are assumed to function properly for radiation shielding in the far wavelength region. In fact, they probably do function reasonably well in that region, and no alternatives are really available at the present.

[16] R. P. Caren, Ibid.

Brief consideration of the numbers quoted above illustrates that most of the incoming energy lies (initially at least) in regions of the spectrum directly accessible to measurement and control, assuming that one has a means of control. Because the blocking of radiant energy is not usually considered from this standpoint, the common approach to control attempts to enclose the cryogenic storage system with low emissivity surroundings, and little concern is typically shown for the spectral characteristics of radiation incident on the thermal protection system. One way of considering the surroundings is to view them as an extension of the thermal protection system.

This situation is aggravated as a spacecraft sits on the ground prepared for launch, since without complicated additional protective measures, gas conductive heat transfer compounds the thermal protection problem. Typically, for many reasons[17], highest boil off penalties are sustained prior to launch as the craft sits ready to go on the pad.

To produce an optical dielectric layer system to reflect radiant energy in the specified wavelength region is a challenge which has not occurred or received consideration in the open literature of film technology. To determine the feasibility of the approach then requires a lengthy inquiry into the present state of the art of optical film design, film materials, and thin film coating technology.

[17] T. M. Flynn, Ibid.

3.1 Background

The beginnings of thin film technology can be traced to the 1930's, when it was observed that surface corrosion of glass altered the optical properties of the material. Techniques were soon highly developed in Germany and elsewhere for increasing the transmission of radiant energy through optical instruments. These antireflection coatings were simple in structure (usually single layer) and widely used. During the following twenty years, the theory of electromagnetic wave propogation through stratified media was developed thoroughly. A fact which emerged from the body of theory was the numerical difficulty and complexity of calculation required to establish the properties of all but the simplest systems. In spite of that, some progress was made in the areas of narrow band reflection and transmission filters. The excellent properties of these devices added further stimulus to the field, which expanded steadily. The development of the electronic digital computer was the turning point in the technology. By use of computers it is possible now to investigate the properties of film structures which could not be calculated by men. Also with computers available interest in the field has turned to many other practical applications, among them broad band reflectors. Novel fabrication techniques have also developed continuously to keep pace with present sophisticated design efforts.

There are many design approaches which might yield broad band high efficiency reflectors, and these will be considered in turn, first. Following that, current manufacturing techniques will be explored.

3.2 Inhomogeneous Optical Reflector Design Methods*

One possibility is to manufacture the reflector of materials so that the refractive index varies continuously in the direction of propogation through the film. Such films are called inhomogeneous, to distinguish them from systems of discrete homogeneous layers, which are the more commonly constructed variety.

In principle, any device which can be manufactured using the discrete layer technology can be made using the continuously varying form. This is true because the latter can be viewed in the limit as a continuous assemblage of a very large number of thin discrete layers. There is, however, a unique set of mathematical problems associated with the theory of inhomogeneous films, which one does not encounter in the discrete case. For this reason, and because of difficulties of manufacture,

*The author is indebted to R. J. Pegis for unpublished notes dealing extensively with this topic.

inhomogeneous films are somewhat neglected in the technology at this point. Because they possess unexplored potential for the present application, it is appropriate to consider briefly what is known of the theory. Since the theory is also relatively obscure, it is hoped the discussion will provide an entry to the scanty literature for the interested reader.

The ordinary way of deriving an expression for the amplitude reflectance of a film is to solve the equations of electromagnetic wave propagation to give the amplitude of the wave electric yield for the forward and backward traveling waves in the medium, denoted E^+ and E^-[18]. The ratio E^-/E^+ can then be related to the reflectance. In the case of a bounded medium whose index n(z) varies only as a function of z, the depth into the medium, the phase thickness in traversing an infinitesimal layer dz is defined

$$dg = \frac{2\pi}{\lambda} n(z)\, dz = \beta(z)\, dz$$

The differential "intrinsic reflectance" is

$$dr = \frac{n(z+dz) - n(z)}{n(z+dz) + n(z)}$$

[18] O. S. Heavens, Ibid.

It should be noted that this form does not represent the differential of the functional form of the total reflectance, because it neglects multiple reflections in the medium. The basic relations for the field amplitudes are

$$dE^{+} = E^{+}(i\,dq - dr) + E^{-}(dr)$$

$$dE^{-} = E^{+}(dr) + E^{-}(-i\,dq - dr)$$

Solving for the indicated field ratio;

$$\frac{d}{dz}\ln\left(\frac{E^{-}}{E^{+}}\right) = \frac{\beta'}{2\beta}\left(\frac{E^{+}}{E^{-}} - \frac{E^{-}}{E^{+}}\right) - 2i\beta$$

and since E^{-}/E^{+} represents the amplitude reflectance,

$$R' = -2i\beta R + \frac{\beta'}{2\beta}(1 - R^{2})$$

This is a Ricatti differential equation, which has no general solution. It yields the reflectance as a function of z and the specified index profile of the film, and must be integrated numerically in general. It can be reduced by substitution to a second order linear differential equation. The physical significance of this equation has been discussed by Schelkunoff[19].

[19] S. A. Schelkunoff Proc Symp Theory of Electromagnetic Waves, Interscience, New York, 1951.

A general review of the theory with many references can be found in the review article of Jacobsson[20].

Although it is not apparent without further consideration, most simple inhomogeneous structures made with real materials and reasonable index profiles have quite low reflectance. However, the promise of high reflectance is held out by some naturally occurring inhomogeneous structures such as hummingbird feathers. Investigations of these have shown that many layers with periodic structure are involved, and that the properties are quite similar to those of the corresponding quarter wave stack, discussed in the next section. Since nowhere has the theory of many continuously varying periodic layers been developed, we have abandoned consideration of inhomogeneous structures for the present application as mathematically infeasible. If further advances in the theory are made, this decision should be reconsidered in the light of the theoretical progress made.

[20] R. Jacobsson, Progress in Optics, V, Wolf, ed. North Holland, New York.

3.3 Homogeneous Optical Reflector Design Methods

Methods of design for homogeneous films have recently been summarized by Pegis and Delano[21]. This summary represents a complete survey of film design methods available thus far, and should be read by anyone interested in the current state of the subject. The most powerful of these methods, the orthonormal optimization method of Grey[22] was used in the present work. The basic software for this purpose has been described by Pegis et al,[23] but extensive modifications of the basic package were necessary to complete this work. We shall briefly describe the intent of the orthonormal technique.

It is usual, and sometimes necessary to describe the properties of a multilayer film device in terms of its physical parameters of construction, that is, layer thicknesses and indices rather than some more abstract representation. As in lens design, the construction parameters may be combined with other quantities to construct a defect function, which expresses the closeness with which the system approaches properties desired for it. A common method

[21] R. J. Pegis, E. Delano, Progress in Optics, Wolf, ed. North Holland New York, 1970.

[22] D. S. Grey J.O.S.A. 53 672 (1963); Ibid 53, 677, (1963).

[23] R. J. Pegis, D.S. Grey, T.P. Vogl, A.K. Rigler, Recent Advances in Optimization Techniques, Wiley, New York 1969.

for improving the system properties (minimizing the defect function) is to vary the construction parameters one at a time, and pursue those variations which yield a lessening of the defect. It has been found in many cases that subsequent variation of parameters can completely undo any system improvements made previously, because of the form of the chosen parameters. This occurs even at the differential level, because of nonorthogonality of the parameter space of the system. Grey suggested a method for the elimination of this difficulty, which depends on transformation to a new, orthogonal coordinate system in the parameter space each time a successful optimization step is made. The method is described in the article of Pegis, et al, to which the reader is referred. The method has been coded into a self sufficient Generalized Orthonormal Optimization Program (GOOP), suitable for use in many areas requiring optimization. Pegis has in turn incorporated GOOP into a proprietary program for film design. Due to the power of the optimization method, this program is probably currently without equal for film design. The design of coatings for the application of interest to this study requires some unusual capabilities, however, and it was necessary to develop a modified program to accomplish the goals of the study. The properties of this program are not fully known at present, and must be explored further before it can be used as a standard design tool in the present application.

Historically, experimentation with wide band reflector designs in the visible part of the spectrum has long been pursued, starting with the work of Baumeister and Stone[24]. However, the term broad band reflector had a much narrower meaning than it does today. Until recently, (excluding trial and error) two basic approaches to the design of such devices have been pursued. The first method is based on the well known properties of the quarter wave stack of alternating high and low index layers[25]. Because these structures, used for laser mirrors, have high reflectance at one wavelength, Turner and Baumeister[26] placed two of these, tuned for slight overlap in the high reflectance region, on the same substrate. Since, however one has the equivalent of two highly reflecting mirrors, separated by a spacer (the adjacent outermost layer of one stack and innermost of the second) the device behaves in similar fashion to a Fabry Perot interferometer, and transmits strongly in the middle of the reflection band. This unforseen effect can be destroyed to some degree by inserting a phase spoiling layer of the proper thickness between the two stacks.

[24]P. W. Baumeister, J. M. Stone, J. O. S. A. 46, 228, (1956).

[25]Military Standardization Handbook, 141, Optical Design, U. S. Government Printing Office, 1962.

[26]A. F. Turner, P. W. Baumeister, Applied Optics 5, 69 (1966).

Using this approach gives a broad band reflector, with the result shown in Fig. 3 . The reflector shown is suitable for use in the short wavelength region of the present application if optimized on the proper substrate.

Other approaches taken to broad band reflector design are of interest to this study. Research at Bell Laboratories has recently been concerned with broad band reflectors for use in optical communications applications. In an article published fortuitously at the commencement of this study, Rigrod[27] has presented a new method of designing broad dielectric reflectors. Although similar in approach to the method of Baumeister and Turner, it rests on the discovery that certain layer structures, having a basic section consisting of more than one pair of layers, and employing two materials having simply commensurate optical thicknesses, exhibit high reflectance properties in regions of the spectrum where low loss dielectrics of very high index are not available. Although the latter condition is not a problem in the infrared, germanium and zinc selenide have a good index ratio ($\sim$2) complying with the first condition. The layer structures are thinner than those of quarterwave stacks, and hence somewhat easier to monitor. The structures of the reflectors proposed by Rigrod are of the form

$$n_0 \left(Y_1/_2 \right)^8 \left(Y_1 Y_2 \right)^4 n_s$$

[27]W.W. Rigrod, Applied Optics 10 1524,(July 1971).

where $$\frac{Y_1}{2} = \frac{\beta_1}{4}\;\frac{\beta_2}{2}\;\frac{\beta_1}{4}$$

and $$Y_1 Y_2 = \frac{\beta_1}{2}\;\beta_2\;\frac{\beta_1}{2}\;\frac{\beta_2}{2}\;\beta_1\;\beta_2\;\frac{\beta_2}{2}$$

where β_1 and β_2 represent the high and low index materials.

The adaptation of this technique to the problem of designing a reflector for the 7-13 μ spectral region, where no previous design has apparently existed, has been successful, and is shown in Fig. 4 . The direct application of the technique yields the reflectors characterized in Figures 5 , 6 . In these two devices, the relative positions of the high and low index layers have been interchanged to observe the performance shift.

In the past, efforts have also been made to enhance the reflectivity of metallic reflectors by overcoating with various materials. Since the scheme of Rigrod works better with increasing substrate index, an effort was made to see if the metal/dielectric composite obtained by using a metallic substrate would result in appreciably improved performance. The result is pictured in Fig. 7 . It is apparent that the performance is little better than the dielectric alone. This, coupled with the already experienced lateral conductivity problems associated with the metals, together with the absolute uncertainty surrounding the optical constants of metals, leads us to abandon this approach entirely for the present.

A brief digression concerning the optical constants of various materials is in order at this point. It is accurate to state that present film design methods far exceed the results of present manufacturing methods in certain areas, due entirely to inadequate knowledge of the optical constants of materials. This is not quite the way one would usually find this fact stated, the inverse statement being more popular. However, the design methods produce exact results, whatever the values used for the constants, while the manufactured product, with exactly specified layer thicknesses, never exactly meets the design performance. Since deposition conditions influence the constants, and layer thicknesses, while closely controlled are never exact, it is usually the manufacturer who deviates most from the recipe, witting or not. The constants of materials are usually measured by reflectance spectrometers of one type or another. These measurements typically can be made to a precision of 1% at present, while 0.1% is required to give results adequate for present design techniques. With the metals, the situation is complicated by their very high absorptance, since the reflectivity changes so rapidly with film thickness that extremely accurate measurements of both quantities are necessary. The result at present is a great disparity in measured results for metals, particularly in the infrared. If this were not enough, the optical properties can be influenced drastically by impurities so that the coater must guard diligently against contamination from handling, improperly trapped vacuum systems, etc. The situation has reached the point where many manufacturers are beginning to use zone refined or otherwise highly purified materials exclusively for their processes.

With dielectrics the situation is not as chaotic, and their properties are somewhat better known, particularly in the infrared, because of military stimulation of the technology. This works in our favor in the present application, and makes the confident prediction of dielectric reflector properties somewhat safer than the prediction of composite properties.

One other promising approach to the design of <u>very</u> highly reflecting broad band coatings has appeared in the literature. The technique, reported by Heavens and Liddell[28], makes use of a coating with layer thicknesses staggered to form either an arithmetic or geometric progression. The geometric forms show broader reflectance, and are classified according as the thicknesses are varied from the outermost layers inward (asymmetric form) or from the central layers outward (symmetric form). An example of each of these forms is shown in Figures 8 and 9. The asymmetric form offers no advantage over our present design and has consequently not been pursued. The symmetric form shows very high reflectance, but has serious transmission leaks toward the long wavelength side. While this design might respond with improved characteristics if subjected to orthonormal optimization, it would have taken much longer than the approach selected. The design should be investigated more fully at a later time.

[28] O. S. Heavens, H. M. Liddell, Applied Optics, 5, 373, (1966).

3.4 Preliminary Optical Reflector Designs for Cryogenic Insulation Applications

The challenge of this study was to determine whether a sufficiently reflective and broad band dielectric substitute for metallizing could be found. We have found one approach for which the reply is affirmative, and two more for which we expect the same answer might result. Before considering these achievements, it is appropriate to consider what properties may reasonably be expected of a dielectric superinsulation reflector. From our own experience with multilayer thin films, and from expert opinions of the wavelength range involved in laminar insulation heat transfer, it is not realistic at all to expect present thin film design technology to span the entire range. In most thermal protection systems, the broadest wavelength range expected is from about 4 to 400 μ, or 100:1. Modern multilayer reflector designs can be made to span 2.5:1 by diligent effort. Hence a single dielectric reflector cannot replace metallized film completely. It is conceivable that a series of dielectric reflectors could, but this would require materials and manufacturing techniques beyond the present state of the art. However, it is possible to obtain a major part of the advantage of these reflectors, using them in the shorter wavelength region, and fabricating them with present day materials and techniques.

An opportunity to demonstrate this lies in a most troublesome insulation operating regime; ground hold. During the time when a rocket sits on the pad ready to launch, the greatest penalties are paid, in terms of poor insulation system performance. There are many reasons for this; a few are insulation compression, hot purging, and generally warmer surroundings than encountered under normal operation. The surroundings of the rocket are radiating at the frequencies characteristic of 300°K black bodies, and the thermal protection systems are immersed in this radiation. From tables of the black body spectral distribution, (see Fig. 10) one finds that the radiated power to be expected at this temperature begins around 4 μ , and that only 15 per cent of the power lies beyond 25 μ . In this environment, lateral conductive effects must be at their worst. This spectral region is accessible to modern dielectric multilayer reflector design and materials technology. Figure 11 shows the properties of some candidate materials in this region. As mentioned earlier, measuring equipment for properties measurement is reliable to about 30 μ , and would be available to assist the effort. The production of a more efficient reflector for use in this spectral region could offer substantially improved system performance in this severe operating environment, and would have further utility in the normal system operating regime. Figure 12 shows the shift in the black body integrated energy fractions as a function of wavelength as the operating temperature is changed. From this it is clear that even in cold systems, an improved reflector working to 25 μ will intercept a substantial fraction of the incident energy. Hence this 2.5 - 25 μ region represents an accessible and useful region suitable for the evaluation of a new approach to superinsulation materials.

In the last two months this has been pursued with success, and interesting results. It has been found that highly reflecting coating designs already exist for the 2.5 to 8 and 13 - 22 μ regions. Furthermore, it has been possible to design a good reflector for the 7 - 13 μ region. The performance of these reflectors is illustrated in Figures 3, 4, and 13. Hence it is feasible to insulate against radiation in this spectral region using dielectric reflectors. However as so often found with technological matters, there are trade offs to be considered. These coatings to be reliably manufactured should have less than about 70 layers each. These do, but three designs can mean three substrates, and surely mean three manufacturing cycles. Consequently, we have designed a thirty-six layer reflector for the 4 - 16 region. This would reduce the requirement for manufacturing to two coatings, and represents an unprecedented wavelength performance range for this type of device, spanning 4 - 1 in wavelength. The performance of this reflector is shown in Figures 16 and 17. While one's initial reaction to the uneven reflectance may be negative, actually it is an excellent reflector when properly used, as we shall show in Section 3.5.

These materials by virtue of their structure exhibit properties as insulation materials that are in marked contrast to those of current materials. Let us discuss these briefly, for they force a change in one's thinking about insulation systems. They have some limitations not present with ordinary metallized film. The optical properties of coating materials available for constructing these devices can change with temperature. In particular, germanium becomes opaque above $50^{o}C$. which would destroy the reflecting properties of the coating above that temperature. The coating will exhibit a reflectivity which varies with angle of incidence for incident radiation. This effect depends on the sensitivity of the individual design, and can usually be reduced to the point where it causes no major difficulty. The 13 - 23 μ reflector shown in Figure 13 has been corrected to eliminate this sensitivity, for example. In the same fashion as a present metallized film, the coatings will exhibit differing reflectivity for radiation of differing polarization incident in a given direction. This effect can be corrected, also if it is serious, but it usually is not of great concern, since unpolarized radiation is a statistical polarization mixture, and the initial calculations done for unpolarized radiation will disclose a low reflectance leading to early discard of the design as unsuitable, if it is sensitive to this effect. A further departure from present metal films lies in the fact that energy not reflected by these coatings will be transmitted, not absorbed in the coating as presently happens. Measured absorptances for dielectric coatings have typically been very low, although the measurements have been done in a limited spectral range, and are normally not measured accurately, since the values are so small. One can normally expect a maximum room temperature absorptances of 0.005 of the incident radiation for dielectrics if good coating practices are followed. Dielectric coatings have been produced with absorptances in the 0.001 region. Note that the 0.005 figure is a factor of 6 below the accepted emissivity of evaporated aluminum, if the dielectric absorptance corresponds rigorously to the emissivity. As mentioned previously that situation is not completely clear, since little attention has been paid to absorptance in dielectrics. Increasing pressure for better coatings is leading to increasing concern with absorptance, and emissivity measurements on some of these materials will eventually be made. If the promise of low emissivity is borne out, these materials will be superior in many respects to present materials. If the energy not reflected is not absorbed, it is consequently not degraded by emission, and hence remains controllable longer as it penetrates the system. Optimization of the insulation system structure must then proceed somewhat differently than it has in the past. From the foregoing, it appears that the dielectric material will be most effective when used as an exterior shield for the metallized film required for the long wavelengths. Using the reflectivity of the dielectric material, it is possible to calculate roughly the radiation environment and hence the power incident on the metallic shields, as a function of the number of dielectric shields. There probably exists some empirical information of the same variety for metallic shields, although not broken down by wavelength.

This brings the discussion to an important segment of measurements which are necessary, to further understanding of these phenomena, and which have apparently not been made. We can list these rapidly, and their lack contributes in major fashion to the noticeably slow progress in laminar insulation system improvement. They are: measurements of monochromatic and directional emissivities for present superinsulations; accurate measurements of the optical constants of metal films in the infrared; measurements of the optical constants of plastic film materials in the infrared (Mylar, Kapton); measurements of the monochromatic and directional emissivities for the dielectric reflectors proposed here.

It should be noted that the lack of optical constant measurements on the plastic film materials in the 2 - 20 μ region has greatly hampered the present investigation, and must be rectified in order to obtain maximum performance from these reflectors. There is some indication in the literature (Fig. 14) that Mylar for example, may undergo index fluctuations in this spectral region. Measurements made recently on Mylar in the region beyond 28 μ [29] shows corresponding fluctuations there. For the present 7 - 13 μ reflector design, an average index value for Mylar of 1.7, interpolated from the near infrared and far infrared measurements, has been used. It should be pointed out here that reflectance measurements

[29] E.V. Loewenstein, D.R. Smith, Applied Optics 10, 577, (March 1971).

made on samples of the reflector coating will immediately disclose spectral regions where this approximate value is incorrect. Further optimization of the design, once the index is known will remove this difficulty. The other coatings for 2.5 - 8 and 13-25 μ region are on BaF_2 and glass, and will have to be optimized for the plastic substrate. This is no point in pursuing this at present, since the correct index is unknown. Additionally, the 13 - 25 design is a patented proprietary coating, and cannot be used in its present form without violating or licensing the patent (Westinghouse Corp.). Optimization of that coating for the plastic substrate should eliminate that problem, however, since the final design should then bear no resemblance at all to the patented version.

3.5 Approaches to the Design of Optical Cryogenic Insulation System

Generally, because of materials inadequacy, the <u>average</u> reflectivities of the dielectric reflectors designed during this study are high, but less than that of aluminized Mylar. For the 24 layer design, for example, the average reflectivity over the range 8 - 13 μ is 92 per cent. However, comparing these materials in this manner overlooks many important advantages possessed by the dielectric material. Briefly, those advantages can be summarized as follows:

Dielectric reflectors are expected to possess low emissivity, and hence can be assembled in deeper stacks than metallized film before the law of diminishing returns sets in. Additionally, no one really knows what happens to the monochromatic emissivity of metals in the infrared. It is known that it fluctuates rapidly, implying that the efficiency of metallic radiation shields is not uniform in this region.

In contrast, the properties of dielectric reflectors can be controlled accurately during the construction, and the reflectance characteristic can be shifted in wavelength at will by ratioing the thicknesses of the reflector layers from some starting point value.

To illustrate graphically the potential of this technique, consider the 36 layer reflector of Figure 16. Notice that the reflectance curve shows many sharp spikes in the shorter wavelength region, and that the transmission spikes are spaced fairly uniformly. Hence if a version of the same reflector were manufactured with its characteristic shifted so that its reflectance peaks correspond to the transmission regions of the first reflector, the combination of the two might yield an excellent reflector. This is the case, as one can see in Figure 18. This reflector, except for one narrow region, is markedly superior to the usual 0.95 reflectivity of aluminized Mylar, and its reflectance exceeds 0.99 over most of the range, hitting 0.9999 in some regions.

This illustrates the production of a nearly ideal optical system element, beginning with less than perfect materials. For a complete system, this material can just be installed in layers, as with a conventional material, or further adjustment of the element characteristics can be performed with a computer to produce a system with specific characteristics in any desired spectral region.

We have not pursued the design of a test system further at this point because of uncertainty regarding the thermal radiation properties of liquid nitrogen, and uncertainty regarding the measured performance of samples of this material. Both of these difficulties will be resolved during a sample fabrication and evaluation phase if the program is continued. From the above, it should be clear that a good insulation material will result from this approach if manufacture is feasible.

3.6 Manufacturing Technology Necessary for the Production of Multilayer Dielectric Insulation Material (DIM)

Multilayer dielectric reflectors for cryogenic insulation applications present an unusual challenge to optical film manufacturing technology. The primary challenge lies in obtaining a coating with good mechanical characteristics. Germanium and zinc selenide are durable materials, so that the only difficulties to be expected will involve:

a. Adhering the coating strongly to the substrate.

b. Adhering the layers strongly to each other.

c. Obtaining amorphous, stress free material in each layer.

d. Obtaining layers of the proper, uniform thickness.

The two materials chosen are widely used in coating fabrication, and a large body of experience is built up concerning their properties and behavior. As far as can be determined at this time, no basic incompatability exists between the two, although zinc selenide has a tendency to crack if excessive stress accumulates in a layer.

There are two differing manufacturing techniques which might be used. They are thermal evaporation, and sputtering.

Thermal evaporation (or deposition) is the name given to the process of heating a material in high (10^{-6} Torr) vacuum until it evaporates, and causing the vapor to condense on an object (the substrate) also located in the vacuum chamber.

Thermal deposition is widely used for the manufacture of optical devices for use in the visible and near infrared portions of the spectrum. It is not widely used yet for components designed for the region 10 μ and beyond. Proof of this is found in the fact dielectric mirrors for use with CO_2 lasers working at 10.6 μ are not yet available, and evaporated gold mirrors on germanium substrates are commonly used instead of the more efficient dielectric structures. The gold coatings do not withstand operation well in this application, and the inability to manufacture dielectric mirrors by this method indicates basic difficulty with the technique in this spectral region. Thermal deposition is the process used for metallizing present superinsulation materials, and some further inferences concerning the suitability of this process can be drawn from consideration of those materials. Thermally evaporated coatings do not adhere well to the substrate in many cases, metallized film being no exception. This is particularly true if the substrate is not scrupulously clean, and the presence of oil films, for example from improperly trapped vacuum pumps, can destroy the adhesion of the coatings. The same effect can destroy the interlayer adhesion as well, although that is less common. Adhesion is usually obtained by heating the substrate, except in the case of metals, which are normally put on cold substrates. Uniformity of coating is a problem familar from metallized film experience, as is sufficient thickness, which never seems to be quite enough.

Difficulty in building thick enough layers is probably a major reason for the lack of 10.6 μ dielectric mirrors.

Taken all together, these problems present a formidable obstacle to development of an efficient DIM manufacturing process using thermal evaporation techniques, and it is specifically recommended that this process be avoided.

Sputtering is a process conducted in a chamber filled to a low pressure with a noble gas, usually argon. An electrode called the target, covered with the material to be deposited is bombarded by gas ions when an electrical discharge is established through the gas. These ions cause molecular fragments of the sputtering material to be ejected from the target. The substrate to be coated is located in a position to intercept and condense the sputtered material.

Because much of the energy of the gas ions can be transferred by collision to the sputtered material, the vapor emerging from the target usually has higher velocities than thermally deposited vapor. As a result, the adhesion of sputtered coatings is tenacious, and there are normally no layer thickness limitations. Layer stress problems can be encountered, and slow deposition rates are observed for certain materials.

Sputtering is an old technique, used to make optical devices for special purposes for many years. In the early history of the technique, it was used exclusively for metallizing. Sputtering processes were found to be very sensitive to the operating conditions, and wide discrepancies appeared in the manufacturing prescriptions published in the early literature.

For this reason, sputtering was gradually replaced by easier and more reliable thermal deposition for optical device fabrication. With continuing slow technological development, culminating in the ability to easily sputter insulating materials using a radio frequency discharge, sputtering has proven to be superior to thermal deposition for many processes. This fact is not widely appreciated and there are still very few specialty firms concerned exclusively with sputtering. Sputtering is used widely at present for integrated circuit fabrication, and would be appropriate for the manufacture of DIM

There appear to be no sputtering rate problems associated with either germanium or zinc selenide, and control of layer uniformity is the only major anticipated problem. The production of batch samples using sputtering should be rapid and straightforward.

When it comes to the manufacture of continuous lengths of this material, a problem appears in that sputtering onto a moving substrate has never been carried out, because no previous requirement has existed. This problem will be faced early in the pilot plant stage of this process, as no way to investigate it is available in present sputtering apparatus. It seems unlikely that the problem will be a serious impediment to manufacture, but it is simply an unknown at this point.

4.0 SUGGESTED PROGRAM FOR FURTHER DEVELOPMENT OF THE DIM CONCEPT

The Statement of Work for the present phase of this contract specifically calls out the reflectance of freshly aluminized Mylar as the measure for success or failure of the optical design effort. In the optical film industry, the measurements of Hass , Fig. 1, made under idealized conditions, are taken as indicative of the best aluminum reflectance attainable. Measurements on aluminized Mylar,[31] (see Fig. 14) illustrate the degradation which occurs in practice as the fabrication is moved from the laboratory to the industrial production chamber. The optical reflector designs of Section 3.4 exhibit reflectance exceeding aluminum reflectance drastically over broad portions of the spectral region, and probably the equal of fully aged (and therefore degraded) aluminizing in the other regions. Apparently no measurements of aged aluminized Mylar have been made, but it is certain that drastic degradation from surface film formation, moisture, and handling does occur. The durability and stability of dielectric coatings compared to metals implies that corresponding aging of the optical reflector would be expected to be much less severe.

[31] Handbook of Military Infrared Technology, U. S. Government Printing Office, 1965.

In light of the foregoing, it is our view that this phase of the investigation has met with remarkable success, considering that this effort represents the first step into a completely new technological area. As indicated in earlier sections, it is not possible to fully develop all promising avenues of investigation during a rapid feasibility demonstration effort. Enough leads in the area were found to indicate that further development building on the results achieved here can ultimately produce a DIM based thermal protection system markedly superior to present systems.

Accordingly, it is appropriate to consider realistically the problems faced in the further development of this approach, and to suggest a careful scheme of procedure from these initial results to the successful demonstration of working hardware. It is important that the suggested plan produce benchmark hardware results with maximum speed, and in an economical fashion, as the expenditure of large sums for development is not justified until the promise of the concept is verified.

4.1 Fabrication of a Limited Quantity of Material

The first obstacle that must be faced is the manufacture of a small quantity of the material. While this is thought to be well within the capability of present coating technology, several problems are expected. Conventional metallizing equipment is not satisfactory for this task, and optical multilayer coating fabricators do not have proper coating system configurations for the production of other than small area batch samples of the material. Since we have also previously indicated that sputtering techniques are the method of choice for fabrication of this material on account of the layer thicknesses involved, we feel that traditional manufacturers of superinsulation materials or multilayer optical devices are completely eliminated from consideration as fabricators for this experimental material.

With sputtering fabricators, the basic problem will be the control of the sputtering target geometry and operating conditions to give sufficient control of sputtering rate parameters. Sputtering-equipment to make batch samples of the material exists now. and it is recommended that batch sample fabrication be initiated at once in order to establish the degree of manufacturing control which must be exercised to successfully produce the material. To measure fabrication progress, one can easily verify the sample spectral performance by requesting N. B. S. spectral measurements on the material. This service is available, inexpensive, and rapid, and would later provide complete justification for expanding the process to the prototype stage.

The construction of a small pilot plant to manufacture continuous lengths of the material should commence at once upon receipt of corroborating measurements on the sample material.

For the proof of the concept in practice relatively small continuous lengths will be required. The plant required for this purpose will not be large, but will require some special features not found on present sputtering systems. No one has yet sputtered material onto a moving substrate, because the requirement has not existed previously. It is expected that conventional sputtering techniques with suitable electrode geometry, or at worst ion gun sputtering,[32] will allow the production of continuous lengths of material with relative ease. Ion gun sputtering replaces the gas discharge ion source of conventional sputtering with a separate ion source, such as a doplasmatron, which is used to bombard the target. This allows precise control of target sputtering rates. Components for either of these systems are off the shelf hardware, and should reguire no development.

4.2 Evaluation of DIM Samples

Since one cannot proceed directly from the design phase to the full scale test of any size tank, because fabrication equipment of the proper geometry does not exist at present, evaluation of samples must serve as the basis upon which

[32] K. I. Chopra, <u>Thin Film Phenomena,</u> Wiley, New York, 1970

the decision to continue hinges. The only alternative to this would appear to be a willingness on the part of the Government to proceed with assembly of the pilot plant on the basis of information in this report. Although that course does not seem prudent until further information is forthcoming, it is a viable alternative if the requirement for this material is urgent.

Trustworthy apparatus for measuring the spectral properties of materials in the middle to far infrared is rare. The N. B. S. Institute for Basic Standards in Gaithersburg, Maryland, has a spectrometer which will make reflectance measurements to 20 μ, at temperatures ranging at least down to $-195^{\circ}C$ (boiling LN_2). This instrument is available for measurements on the DIM samples at a nominal charge per sample, with a ninety day maximum response time. This appears to be the only instrument in the United States suitable for the purpose, and its use should indicate completely the progress of the fabrication effort. In the absence of any alternative which does not jeopardize the final success of the development program, it is recommended strongly that spectrometer measurements be used as guides through sample fabrication, with pilot plant assembly begun immediately upon verification of manufacturing process parameters.

4.3 Cryogenic Tank Testing of DIM Systems

Cryogenic tank testing of this material is the goal toward which all the effort is directed. Since it has not yet been possible to fully assess the unusual potential capabilities of this material, only some general outlines of a tank test program can be drawn at this point.

Considering the somewhat unexpected results discovered during this final study; i.e. cryogenic liquids are prone to absorb narrow spectral regions of infrared radiation, and DIM reflectors are presently restricted to somewhat narrow spectral regions unless produced in large complex arrays, it is difficult to presently propose a fair comparison tank test between a normal metallized film system and a DIM system. Again from the results of this study, it seems absolutely certain that DIM offers some unique advantages for radiation shielding of cryogens. The foregoing makes it appear useful immediately as a weight saving supplement to present systems and probably as a major system component when the development of better reflector designs can be carried further.

While it is now feasible to produce the complex multilayer coating arrays required to replace much of the metallized film used in this application (some extremely complicated thin film devices have been successfully used in space in the past), the expense of such an undertaking is not justified until some experimental results whose significance is properly understood can be generated, and until these same experimental results can be used to benchmark the approaches suggested earlier by the theoretical understanding of the dielectric structures.

Additional time and further study are going to be required following the fabrication and evaluation of samples, in order to consider fully the many system trade offs generated during this present study phase, and to establish a fair test of the concept. To fully illustrate the superiority of these materials, the best operational test conditions should be selected in light of experimentally determined material properties. It is a waste of effort to give a detailed account of the structure of the completed insulation system if the material cannot be made. The best one can do, given our slowly receding ignorance, is to suggest, as we have in Section 3.5, those structures which appear promising for this purpose at present.

To keep the economics within reason, the test bed tank used for this investigation should be a small tank, with the simplest support/penetration system possible to avoid confusion arising from those sources. The proper cryogen for test purposes must be determined following experimental verification of the manufacturing techniques. Unless unforseen advances can be made, the manufacturing effort must have reached the pilot plant stage before tank tests become feasible.

4.4 Continuing Evolution of Reflector Design

During this feasibility demonstration effort, some promising design techniques for infrared reflectors were disclosed. Continuing effort should be expended to make certain that nothing of value in these has been overlooked. In addition, continuing design effort should be directed at improving and altering for other purposes the properties of the reflectors successfully designed during this phase of the study. This unexplored technology undoubtedly contains other unforseen applications to spacecraft thermal control and power generation.

5.0 APPENDIX I

BIBLIOGRAPHY OF REFERENCES

OPTICAL PROPERTIES OF

SUPERINSULATION AND ITS COMPONENT

AT CRYOTEMPERATURES

CHARACTERISTIC CODING DESIGNATIONS for CRYOGENIC LITERATURE

Categories

A-1: Books, Reviews, Surveys, Bibliographies, Proceedings, etc.
A-2: Properties of Solids
A-3: Properties of Fluids
A-4: Solid State, Theoretical, Phenomena, Basic Physics, etc.
A-5: Cryogenic Techniques, Tricks, Unique Methods, Unusual Procedures, etc.
A-6: Cryogenic Processes, Heat Transfer, Purification, Fluid Flow, Liquefaction, Safety Procedures, etc.
A-7: Laboratory Equipment and Instrumentation
A-8: Cryogenic Equipment
A-9: General Interest Literature, News, Management, Programs, Accidents, Miscellaneous

Language

B-1: English, B-2 French, B-3 German, B-4 Dutch, B-5 Italian, B-6 Japanese, B-7 Russian, B-8 Spanish, B-9 Other

Cryogenic Interest

C-1: Cryogenic Temperature Range (0 to 130°K where not specifically designated in C-4 through C-7 below)
C-2: Cryogenic Interest but not in Cryogenic Temperature Range (except where designated C-8)
C-3: Not of Direct Cryogenic Interest
C-4: Below 1°K
C-5: 1 to 10°K
C-6: 10 to 50°K
C-7: 50 to 130°K
C-8: 130 to 300°K

Form of Data (Omitted where not pertinent)

D-1: Numerical Data Included
D-2: No Data
D-3: Graphical Data Only

Type of Article (Omitted where not pertinent)

E-1: Experimental, Experimental and Theoretical, Original Work
E-2: Review Article, Compilation, Correlation, Discussion
E-3: Theoretical Only, No Specific Data Given

Availability of Document (suggested source)

F-1: Cryogenics Division
F-2: National Bureau of Standards
F-3: Clearinghouse for Federal Scientific and Technical Information (CFSTI)
F-4: U.S. Government Printing Office
F-5: Defense Documentation Center (DDC) or NASA Scientific and Technical Information Facility
F-6: Technical Libraries Generally (Published Literature)
F-7: Technical Libraries - Special (Foreign Literature - Special Periodicals)
F-8: Company Bulletins and Reports (Universities, Research Labs., etc.)
F-9: Other (Patents, Theses, Translations, etc.)

Form of Document

G-1: Published - Open Literature, Journals, etc.
G-2: Books, Proceedings
G-3: Company Periodicals (includes University, Foreign Gov't, State Institutions, etc.)
G-4: Government Periodicals (U.S.)
G-5: Company Reports, Private, Public, Gov't Contract (includes Foreign Gov't Reports)
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SUBJECT INDEX

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THERMAL RADIATION, THICKNESS, VACUUM, FREQUENCY EFFECT,
ENERGY EXCHANGE, TUNNEL EFFECT, INTERFERENCE, *INSULATION,
VACUUM INSULATION, MULTILAYER INSULATION,

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*INSULATION, MULTILAYER INSULATION, RADIATION SHIELD, HEAT LOSS,
*HEAT TRANSFER, SPACE APPLICATION, CALCULATION, EMISSIVITY,
A2 B1 C6 D3 E3
*EMISSIVITY, *ALUMINUM, *GOLD, *SILVER, SURFACE COATING,
RADIATION SHIELD,

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SURFACE TREATMENT, RADIATION HEAT TRANSFER, *STORAGE, ANALYSIS,
SOLAR REFLECTOR, BOIL OFF,

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FEEDTHROUGH, MULTILAYER INSULATION, INSULATING SUPPORT, *STORAGE,
THERMAL RADIATION SHIELD, *LIQUID HYDROGEN, SIZE EFFECT,
RADIATION HEAT TRANSFER, THERMAL CONTACT RESISTANCE,
A2 B1 C1 D1 E3
*THERMAL CONDUCTIVITY, RADIATION SHIELD, *INSULATION, EMISSIVITY,

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*INSULATION, FOAM, GLASS REINFORCED PLASTIC, URETHANE, ALUMINUM, *LIQUID HYDROGEN, EMISSIVITY, BOILOFF, CENTAUR, *HEAT TRANSFER, *LOX, PANEL, TANK, THERMAL RADIATION SHIELD,
A2 B1 C6 D1 E1
*ALUMINUM, *EMISSIVITY, PAINT,

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A6 B1 C7 D1 E1
*INSULATION, *HEAT TRANSFER, MULTILAYER INSULATION,

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A3 B1 C6 D1 E1
*EMISSIVITY, PAINT,
A2 B1 C6 D1 E1
*FOIL, *ALUMINUM, MYLAR, *EMISSIVITY, *COPPER, STAINLESS 304,

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*INSULATING SUPPORTS, JOINING, CONSTRUCTION TECHNIQUE,
A2 B1 C8 D1 E1
*INSULATION, *EMISSIVITY, SUPERINSULATION, PRESSURE EFFECT,
MYLAR, POLYIMIDE, ALUMINUM, MOON, TEST RESULTS,
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*HEAT TRANSFER, CONDUCTION, THERMAL RADIATION, STAINLESS STEEL,
EMISSIVITY, TEMPERATURE PROFILE, RADIATION,

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RADIATION HEAT TRANSFER, *INSULATING SUPPORT, *INSULATION,
*LIQUID HYDROGEN, BOILOFF, FIBERGLASS, TITANIUM, STAINLESS STEEL,
ALUMINUM,
A2 B1 C6 D1 E1
*INSULATION, *OPTICAL PROPERTY, REFLECTANCE, RADIATION SHIELD,
MULTILAYER INSULATION, *EMISSIVITY, FILM, POLYESTER, COATING,
A2 B1 C6 D1 E1
*THERMAL CONDUCTIVITY, *BERYLLIUM, *ALUMINUM, *TITANIUM,
FIBERGLASS, *STAINLESS STEEL,

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EMISSIVITY, CALCULATION, ALUMINUM, CHROMIUM, BRASS, NICKEL,
SILVER, GOLD,

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A2 B1 C7 D3 E1
*EMISSIVITY, *FILM, MYLAR, ALUMINUM, SURFACE COATING, POLYIMIDE, EMITTANCE, GOLD, *COPPER, METALLIZED, *COMPOSITE,

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A2 B1 C6 D1 E1
MULTILAYER INSULATION, MYLAR, ALUMINUM, SURFACE COATING, FOIL, *EMISSIVITY, EMITTANCE, *INSULATION,

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A2 B1 C8 D1 E2
*INSULATION, MULTILAYER INSULATION, *OPTICAL PROPERTY, *MYLAR, *ALUMINUM, BOROSILICATE GLASS, FIBER, *EMISSIVITY, TRANSMITTANCE, REFLECTANCE,

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A7 B1 C7 D1 E1
*OPTICAL DEVICE, EMITTANCE,

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GLASS FIBER, PRESSURE EFFECT, *INSULATING SUPPORTS, *CRYOPUMPING,
*CRYOGENIC CONTAINER,
A2 B1 C5 D1 E2
*OPTICAL PROPERTY, ABSORPTION, SKIN EFFECT, *COPPER, *ALUMINUM,
*TIN, *LEAD, *BRASS, *CADMIUM, *CHROMIUM, *GOLD, *IRON,
*NICKEL, TIN ALLOY,
A2 B1 C1 D1 E2
*OPTICAL PROPERTY, ABSORPTIVITY, *ALLOY, *ZINC, *SILVER,
MANGANIN, MONEL, CHROMIUM-NICKEL STEEL,
A2 B1 C1 D1 E2
*INSULATION, *THERMAL CONDUCTIVITY, *THERMAL EXPANSION, MOISTURE,
PARTICLE SIZE, FOAM, POWDER,
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CONDUCTION, SUPERINSULATION, EMITTANCE, THERMAL RADIATION,